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Studies in Soil Colloids, Part I

Base exchange and soil acidity

BY

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Late Physical Chemist, Imperial Institute of Agricultural Research, Pusa

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It is well known that colloidal materials of different soils differ markedly in chemical composition.[1] However, a number of physico-chemical reactions show a remarkable similarity. We may suppose that a soil colloidal particle is made up of a number of molecules, each composed of a simple positive radicle and a complex negative one. The positive radicle is directed towards water in a soil suspension and largely determines the physico-chemical properties of the colloid.

From the agricultural and practical point of view, we are concerned mostly with the positive radicle, and as a first approximation we can leave out of account the rest of the molecule, the nature of which is only revealed by such drastic treatments as heating with strong hydrochloric acid, etc.

On coming in contact with water, the molecules of soil colloids will have a tendency to dissociate. However, the comparatively light electropositive radicle, being associated with a heavy insoluble negative group, will be unable to leave it. Thus we shall have insoluble or colloidal particles floating about in water, each carrying a number of ions sticking out as it were from the surface. These surface active ions will be capable of entering into chemical reactions, so that if they come under the sphere of influence of a higher concentration of a different electropositive ion they will have a tendency to be replaced by the latter. Also, if the colloidal particles are suspended in a solution containing ions that are different to those on the surface, then a sort of dynamic equilibrium will be set up characterized by the number of ions in solution taking the place of those on the surface, and *vice versâ*. The state of equilibrium will depend on the nature of ions, their concentration and the temperature of reaction.

In effect, to use the nomenclature of Michaelis, in soil colloids we are dealing with " acidoids " if the surface active ion is H, and " saloids " if it is any other electropositive ion. This is the widely accepted view in one form or other.[2]

[1] P. L. Gile. Nature of the Colloidal Soil Material. *Colloid Symposium Monograph*, Vol. III p. 216 (1925).

[2] Page, H. J. The Nature of Soil Acidity. *Trans. 2nd Commission Intern. Soc. Soil Sci. Groningen.* Vol. A, p. 232 (1926).

If this theory is substantially correct, then we should expect the following reactions and phenomena to be characteristic of soil colloids :—

(1) It should be possible to replace all the surface active ions by H and this amount should be a characteristic constant for a given soil colloid.

(2) The " acidoid " thus produced should show most of the reactions characteristic of true acids.

We shall therefore see how far these expectations are realized from experimental facts.

We may represent the action of an acid, say HCl, on the molecule of a soil colloid as follows :—

$$AX + HCl \rightleftarrows HX + ACl.$$
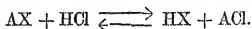
Where A is any diffusible electropositive ion and X the heavy non-diffusible electro-negative radicle. Since it is a reversible reaction, the accumulation of ACl will favour the back reaction ; obviously, if one of the products of reaction, that is, ACl, be continuously removed, we should come to a stage when the whole of AX has been converted into HX " Acidoid." Conversely, we can again convert HX into AX by exhaustive treatment with ACl, continuously removing the HCl thus produced until the reaction has reached a well-defined end point, when no more HCl comes in solution.

Experiments with a number of soils showed that when a soil is exhaustively treated with $\frac{N}{20}$HCl and washed free from the excess of acid, the *amount of H retained by it is a characteristic constant for that particular soil.* Further, a part of this H can be replaced by any other electropositive ion by the exhaustive treatment of its neutral salt. The total free acid thus produced, if titrated to phenolphthalein, is found to be chemically equivalent to the other ion taking the place of the H ions. That ions replace one another in equivalent amounts has already been shown by Gedroiz.[1]

The results with a number of soils in which Ba and H mutually replaced each other are recorded in Table I.

In every case 10 gram of air dry soil was used. This was treated with $\frac{N}{20}$ HCl continuously in a special percolating cylinder [2] until the filtrate showed no reaction for Ca ions. It was then washed with $\frac{N}{5}$ BaCl$_2$ in 100 c.c. lots ; each lot titrated with $\frac{N}{10}$ baryta using phenolphthalein as indicator. When free from acid, it was washed with water until free from Ba. It was again washed with $\frac{N}{20}$ HCl until all the Ba was displaced and determined in the filtrate.

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[1] Gedroiz. K. K. The absorbing capacity of the soil and the zeolitic bases of the soil. *Zhur Opit. Agron.*, 17, p. 472 (1916).
[2] Puri, A. N. A new percolating cylinder and some of its uses. *Agri. Jour. India*, Vol. XXIV, No. 6.

TABLE I.

Showing the equivalence of H and Ba in the exchange complex.   Milli equivalents per 10 grams of soil.

Soil											H(phenolph-thalein)	Ba
1. Gonhri (Pusa)		.	.	.	.	.	.	.	.	.	0·511	0·566
2. Rendzina	.	.	.	.	.	.	.	.	.	.	1·373	1·558
3. Hoos Fallow (Rothamsted)		.	.	.	.	.	.	.	.	1·357	1·418
4. Gokak .	.	.	.	.	.	.	.	.	.	.	1·676	1·73
5. Dharwar	.	.	.	.	.	.	.	.	.	.	4·588	4·73
6. Burma soils D_2	.	.	.	.	.	.	.	.	.	.	0·235	0·225
7. ,, ,, D_3	.	.	.	.	.	.	.	.	.	0·22	0·233
8. ,, ,, D_4	.	.	.	.	.	.	.	.	.	0·50	0·542
9. ,, ,, D_5	.	.	.	.	.	.	.	.	.	0·205	0·32
10. ,, ,, D_6	.	.	.	.	.	.	.	.	.	1·24	1·36

Considering that acidity determination involved at least half a dozen titrations in the presence of Al salt, the agreement between the H and Ba is fairly close.

It may be pointed out that in every case a part of the acid displaced reacted with the soil complex dissolving Al and Fe.   It is assumed that phenolphthalein gives an end reaction in which chlorides of Al and Fe figure as acid.   Titrations done in the presence of methyl orange as indicator always give low figures.

That Al and Fe appear as a result of side reaction and hardly any as a result of direct exchange is shown by the fact that, however long a soil be washed with $\frac{N}{50}$ HCl, there always appears a certain amount of Al and Fe and *longer washing does not increase the amount of replaceable H but rather decreases* it slightly, due to the breaking up of the exchange complex.   On the other hand, all other exchangeable bases can be completely removed by washing with $\frac{N}{20}$ HCl.   This dissolution of the exchange complex by acids takes place according to the well known adsorption law of Freundlich.   So that if a soil that has been completely desaturated by treatment with $\frac{N}{20}$ HCl is shaken with increasing concentrations of dilute HCl and then titrated after filtration with standard alkali using methyl orange as indicator, the decrease in acidity (*i.e.*, the amount of acid reacted with the soil), when plotted against the equilibrium concentration, follows a curve which may be approximately fitted into

an exponential one (Fig 1), but the interesting point about it is that the maximum value for the Al coming in solution is approximately equivalent to the " acidoid " (H_1) content of the soil.

Dissolution of Al from Completely unsaturated soils by dil. HCl (5 G·ms soil)
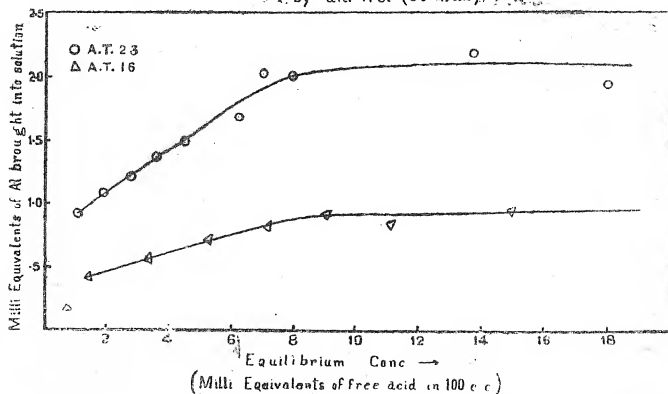
It is also noted that this value is reached when the concentration of HCl is about N/10, indicating that if a completely unsaturated soil is shaken with N/10 HCl, then the amount of Al coming in solution would be equivalent to its " acidoid " (H_1) content, (*i.e.*, only the H replaceable by neutral salts).

Thirteen soils and clay fractions ($>$·001 mm. diameter) were examined by this method, and, as will be clear from Table II, the agreement is fairly close.

TABLE II.

Showing the equivalence of Al dissolved by $\frac{N}{10}$ HCl and the (H_1) " acidoid " content of completely unsaturated soils.

Soils	Acidoid (H_1) Milli equivalents per 10 grams	Al Milli equivalents per 10 grams
A. T. 1	1·64	1·85
A. T. 2	0·90	0·88
A. T. 3	0·86	0·92

<div align="center">

TABLE II—*contd.*

Showing the equivalence of Al dissolved by $\frac{N}{10}$ HCl and the (H_1) " acidoid " content of completely unsaturated soils—contd.

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Soils	Acidoid (H_1) Milli equivalents per 10 grams	Al Milli equivalents per 10 grams
A. T. 15	0·65	0·40
A. T. 16	1·70	1·68
A. T. 17	1·50	1·20
A. T. 23	4·40	4·00
A. T. 24 A	4·40	4·80
Clay fractions.		
1. F. A.	2·30	2·20
2. F. A.	3·10	2·20
3. F. A.	3·20	3·30
4. F. A.	3·60	3·52
6. F. A.	2·92	3·52

Nos. 2 and 6 were clay fractions separated from soils rich in humus.

It might be mentioned that it is necessary for all these studies that the soil used should be desaturated as completely as possible. The easiest method of finding whether a soil is actually in a state of complete unsaturation is to shake about 10 grams of the soil with 100 c. c. of $\frac{N}{10}$ HCl for a couple of hours and then filter. An aliquot portion of the filtrate is titrated against standard alkali *using phenolphthalein* as indicator. If the soil is completely unsaturated, there will be a very small decrease in titratable acidity.

It is difficult to see at present what the exact significance of this agreement between the values of Al dissolved and the (H_1) acidoid content can be, but the point is worthy of note in connection with the toxicity of Al salts towards plants. It appears that two soils having the same degree of unsaturation might bring different amounts of Al in solution. The fact might explain why certain soils with a low pH value can support plants while in others the same pH proves toxic. However, no definite conclusions can be drawn until the toxicity of Al salts has been studied with particular reference to the absorbing capacity of soils by pot experiments and field trials.

It is intended to study this not only from the point of view of the question raised above, but to gain information with regard to the bigger problem of the nature of

toxicity due to soil acidity, *i.e.*, whether the toxicity of H ions is direct or indirect through their ability to bring about the dissolution of Al.

We have seen that if a soil in which the exchange complex is in the form of " acidoid," is continuously treated with $BaCl_2$, the reaction proceeds to a well defined limit, but we cannot presume that this corresponds to the conversion of the whole of the " acidoid " into its Ba " saloid," for we know that the Ba saloid thus produced is capable of yielding titratable acid when treated with a hydrolysed salt like Ba acetate. This point will be referred to later. However, for convenience, we may refer to the hydrogen ions that can be displaced from the soil acidoid by the action of neutral salts as H_1. This being understood, we can speak of the H_1 content of a soil, knowing also that the maximum H_1 content of a soil is a characteristic constant for that soil.

Having seen that every soil can develop a fixed amount of acidoid that can be easily determined by the exhaustive treatment with $BaCl_2$ (H_1 content), a number of soils were thus converted and several reactions characteristic of true acids were studied on these fully unsaturated soils. These were as follows :—

    (1) Inversion of sucrose.

    (2) Hydrolysis of Ethyl acetate.

    (3) Reaction with a carbonate and determination of CO_2 evolved.

    (4) Reaction with a hydroxide and determination of the hydroxide neutralized.

    (5) Reaction with a sulphide and determination of H_2S evolved.

    (6) Reaction with ammonia.

    (7) Reaction with amines.

    (8) pH determination of suspensions.

    (9) Reaction with neutral salts.

  (10) Heat of neutralization.

These are dealt with below :—

(1) Inversion of Sucrose.

Inversion of sucrose by soil has been studied before by Tacke and Suchtig,[1] Hanley,[2] and more critically by Rice and Osugi,[3] who showed that a suspension of acid soil inverts several times as much sugar as an extract of the same soil. They also found the inversion greater when a soil was kept in suspension by continual shaking than when it was permitted to settle out in the flask. Consequently they found no quantitative relation between the pH value of the suspension and sugar inversion, and concluded that the latter should give a better measure of soil acidity than the former.

[1] Tacke, B., and Suchtig, H.   *Landw. Jahrb. Bd.*, 41, pp. 717-754 (1911).
[2] Hanley, J. A.   The humus of acid and alkaline peats.   *Jour. Agri. Sci.*, Vol. 6, pp. 63-76 (1914).
[3] Rice, F. E., and Osugi, S.   The inversion of cane sugar by soils and allied substances and the nature of soil acidity.   *Soil Sci.*, Vol 5, p. 333 (1918)

The experimental technique employed by all the previous workers left much to be desired. Tests were carried out at temperatures as high as 83-85°C. without a thermostatic control. Apart from that, any results obtained under those conditions may not be applicable to the behaviour of the soil at lower temperatures.

The experiments on the sucrose inversion, in this case, were carried out in a thermostat kept at 40° C. (±·05). In every case 8 per cent. sucrose was used (8 grams of sucrose to 100 c. c. of water). The time of contact was four hours. The suspension was shaken after every ten minutes or so. At the end of four hours the mixture was at once cooled to about 10°C. and filtered. The amount of inversion was determined by adding 25 c. c. of the filtrate to a known amount of Fehling solution and completing the reduction by a standard 0·1 per cent. invert sugar solution. The concentration of the solution was thus known in terms of the standard invert sugar. For purposes of comparison, it gave satisfactory results as the experimental conditions were kept the same in every case. Direct titration was discarded on account of the very low inversion in some soils.

Six soils from various parts of the world were employed for the study of sugar inversion. The H_1 content of each determined by exhaustive treatment with $BaCl_2$ is recorded in Table III.

TABLE III.

H_1 content of various soils used for sugar inversion experiments.

Soil	Milli-equivalents of H_1 per 100 grm. of soil
1. Akola (India)	45·30
2. Dharwar (,,)	47·04
3. Michigan (U. S. A.)	17·10
4. Rendzina (Tschechoslowkei)	16·39
5. Roos fallow (Rothamsted)	15·30
6. Gokak (India)	14·30

The soils were dried over H_2SO_4 before use. Increasing amounts of each of the first three soils were added to 100 c. c. of water and left overnight. Next morning the bottles were placed in the thermostat at 40°C. for about half an hour, 8 grams of pure sucrose was then added to each bottle and the mixture left for four hours with frequent shaking at the end of which it was immediately cooled and filtered under suction. A clear solution was always obtained.

The results are given in Table IV and plotted in Fig. 2, the abscissa being the amount of H_1 in milli-equivalents and the ordinate percentage of sugar inverted.

TABLE IV.

Sugar inversion by various soils.

	Weight of soil grams	H_1 content Milli-equivalents	Sugar inverted per cent.
Akola	1	0·453	0·33
	2·5	1·132	0·47
	5	2·264	0·65
	10	4·53	0·98
	15	6·794	1·49
	20	9·06	1·86
	25	11·324	2·6
	30	13·588	3·5
	35	15·854	3·73
	40	18·12	4·30
Dharwar	1	0·4705	0·4
	2·5	1·176	0·46
	5	2·352	0·585
	10	4·704	1·10
	15	7·056	1·605
	20	9·408	2·14
Michigan	1	0·171	·245
	2·5	0·4275	0·365
	5	0·855	0·495
	10	1·71	0·675
	15	2·565	0·845
	20	3·42	0·945
Rendzina	20	3·278	0·785
Hoos fallow	20	3·06	0·585
Gokak	20	2·86	0·66

It will be seen that all the results with different soils, when considered in terms of the H_1 content, fall approximately on one curve. This is a remarkable fact and

seems to show that " acidoid " in different soils possess very nearly the same " dissociation " or " activity " coefficient.   In other words, if soluble, they would all be equally " strong " or equally " weak " acids.

Through a strange coincidence, the inversion by acetic acid is almost of the same order as that by the " acidoid " in equivalent concentration.   The results with acetic acid are also plotted on Fig. 2 and show a very good agreement up to a certain point, after which there is a systematic deviation.
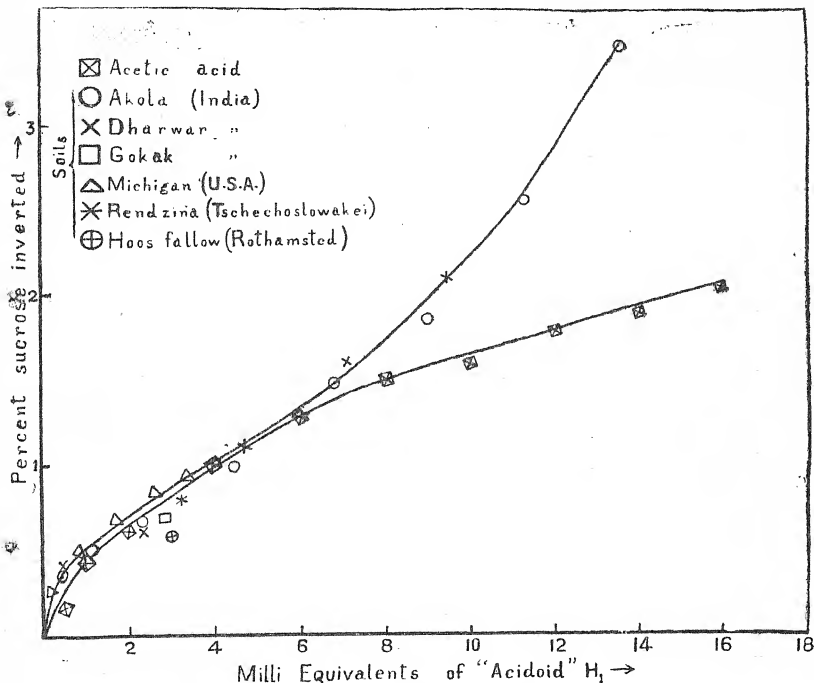

Fig. 2.

A moment's reflection will show that this deviation is not quite unexpected. Increasing concentrations of acetic acid mean that increasing amounts of the acid

are being compressed in the same volume, so the ionisation must alter appreciably as the solution gets stronger, whereas in the case of soils increasing amounts were added to 100 c. c. of water and there was not so much overcrowding of the molecules as in the case of acetic acid.

8 per cent. sugar solution was chosen, because at higher concentrations there is likely to be some absorption of water or a weaker solution of sugar, leaving the rest more concentrated.[1]

Having established that sugar inversion is proportional to the amount of H_1*, we could use this method for obtaining a sort of titration curve for the same. 100 c. c. of Ba (OH)$_2$ solution of various concentrations were added to 20 gram portions of Akola soil and the mixture left for three days with occasional shaking. Sugar inversion was then studied by adding 8 grams of sucrose to each as usual. The results plotted in Fig. 3 show the characteristic shape of titration curve of a weak acid. The curve meets the abscissa somewhere about the point of neutralization of the H_1.

Next another method of neutralizing the "acidoid" was chosen. The same soil (Akola) was partly depleted of its surface active hydrogen by replacement with Ba. This was accomplished by treating 25 grm. lots of the soil with varying quantities of $\frac{N}{5}$ BaCl$_2$ solution in percolating cylinders. The acid displaced was titrated and the excess of BaCl$_2$ removed by washing.

Thus were obtained soils containing varying amounts of "acidoid" and its Ba "saloid." Inversion was studied with this series. These results are also plotted in Fig. 3.

The gradual introduction of Ba in the exchange complex being expressed in terms of the acid displaced which was titrated against the same baryta used for the other titration curve, the two sets of observations are, therefore, strictly comparable. It was expected that the two curves will be almost superimposed, but they seem to follow different courses, however, the neutralization point, characterized by the stoppage of sugar inversion, falls approximately on the same point.

That the neutralization of "acidoid" with Ba (OH)$_2$ may be slightly different from its "saloid" formation through neutral salt reaction was also shown by the fact that the filtration of the first series of soils was very difficult, while the second set was not so. This was quite a frequent observation.

It is quite likely that the mechanism of the two reactions may be something as follows :—

$$\text{I} \qquad X{<}^{H}_{H} + Ba{<}^{OH}_{OH} \longrightarrow X{<}^{H—OH}_{H—OH}{>}Ba$$

$$\text{II} \qquad X{<}^{H}_{H} + Ba{<}^{Cl}_{Cl} \longrightarrow Ba\ X + 2\ HCl.$$

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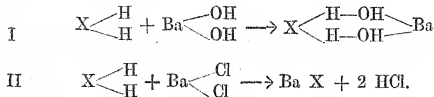
[1] Puri, A. N. The hypothesis of unfree water in soils. *Agri. Jour. India*, Vol. XXIV, No. 5
*Water extract of these soils showed hardly any inversion under the same set of conditions.

Titration Curves for Akola Soil, by Sugar inversion

(The Soil Contained 45·3 Milli Equivalents of H "acidoid" per 100 Grms)

I CC's of $\frac{N}{10}$ Ba (OH)₂ added to make 100 c.c of sol,
   (20 Grms of soil added to it)

II Equivalent amount of H replaced by Ba
    (by treatment with Ba Cl₂) →

Fig. 2.

Percent sucrose inverted →

According to this hypothesis, the first reaction would give a highly hydrated and consequently gelatinous compound which agrees with the fact that it is difficult to filter. Further, it is conceivable that such hydration might reduce the extreme range of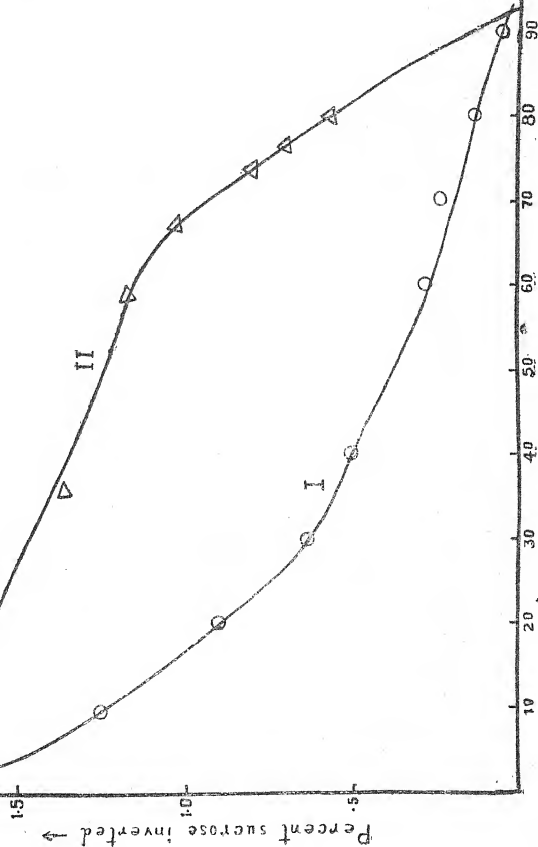 movement of the rest of the surface active hydrogen ions by reducing the permeability of the well known "Double Layer," thus a greater reduction in the amount of sugar inverted might be expected in the first case than in the second by the introduction of the same quantity of Ba in the Exchange complex.

It is also possible that this difference may be due to the fact that a part of the free HCl produced in the second reaction is probably not washed away completely and since it is a very active acid a small amount might cause the extra inversion observed. It might be mentioned that pH values of the suspensions in the second case were always lower than those in the first.

(2) Hydrolysis of Ethyl acetate.

Parker and Bryan [1] compared the inverting and hydrolysing power of acid soils with sucrose and ethyl acetate, respectively, both with suspension as well as water extract. They found that hydrolysis of ethyl acetate was greatest in water extract and least in soil suspension; on the other hand, sugar inversion was greatest in soil suspension and least in water extract. They concluded, therefore, that acid silicates catalyze the inversion of cane sugar and not the hydrolysis of ethyl acetate which is brought about only by a higher concentration of H ions in the mass of the soil solution.

The above authors considered the possibility of a partial neutralization of the acetic acid by acting on the minerals of the soil, but did not think it would account for the extremely small hydrolysis brought about by soil suspensions.

Considering the large amount of exchangeable bases even in acid soils, the possibility is by no means remote that almost the whole of the acid would be used up by the soil when the hydrolysis takes place in the presence of suspension. Besides any small quantity of the acid produced in the first instance would be at once removed from solution and would not be available for hastening the reaction by auto-catalysis. As will be seen presently, the supposed difference disappears when completely unsaturated soils are used, as all the acetic acid produced in this case remains in solution, there being no exchangeable bases in the soil to react with it. The following experiments were performed :—

(a) In this case, hydrolysis of Ethyl acetate was studied with 5 soils and 5 clay fractions separated from 5 different soils. The soils and clay fractions were all completely unsaturated as in the case of sugar inver-

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[1] Parker, F. W., and Bryan, O. C. Soil acidity as measured by sugar inversion, the Truog test and the Hydrogen ion conc. and its relation to the hydrolysis of Ethyl acetate. *Soil Sci.*, 15, p. 99 (1923).

sion experiments. 5 c. c. of Ethyl acetate were dissolved in 100 c. c. of water, to this 20 grams of soil or 5 grams of clay fractions were added. The bottles were shaken during 48 hours (shaking stopped from 10 P.M. to 6 A.M.). The suspension filtered after this, a clear filtrate was obtained in all cases, as the acetic acid liberated, flocculated the soil to a certain extent. 25 c. c. of the filtrate titrated with $\frac{N}{10}$ alkali to phenolphthalein. The results plotted in Fig. 4 show a curve similar to that of sugar inversion, and likewise all the points fall on one curve.

Fig. 4.

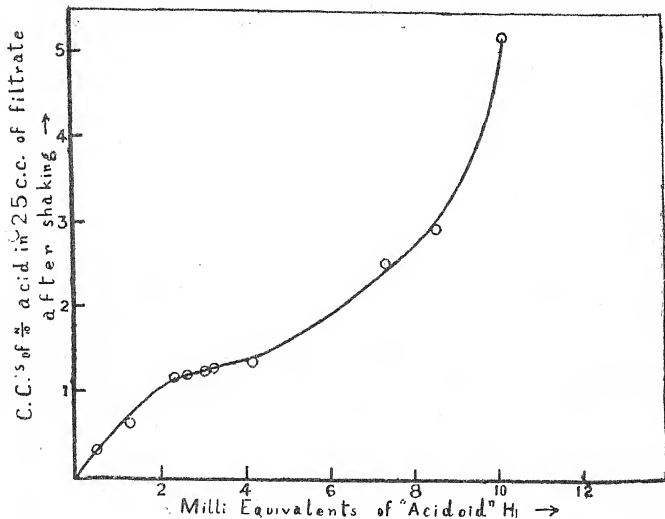(b) Varying amounts of a 4 per cent. clay suspension were made up to 100 c. c. with water and 5 c. c. of Ethyl acetate (total vol. 100 c. c.). Parallel to this series, varying strengths of acetic acid containing 5 c. c. of Ethyl acetate were shaken ; so that for every concentration of clay suspension there was a corresponding concentration of acetic acid containing the same amount of acid in 100 c. c. as the acidoid (H_1)

in the suspension. The results plotted in Fig. 5 show a close similarity between the two sets of values.

As in the case of sugar inversion, the curves show a greater divergence towards the latter part, because in the case of clay suspensions since the total volume was 100 c. c. the amount of water gradually decreases as the concentration of clay increases, so that we are actually dealing with a stronger solution, and 25 c. c. of the filtrate would naturally contain more of the product of hydrolysis than in the case of acetic acid solutions.

As in the case of sugar inversion, the water extract of soils showed just about the same hydrolysis as pure water. For instance, 100 c. c. of the water extract from the clay suspension gave 0·12 c. c. of hydrolysed acid per 25 c.c. against 0·2 c.c. obtained with re-distilled water.

(3) REACTION WITH A CARBONATE AND DETERMINATION OF CO_2 EVOLVED.

The decomposition of $CaCO_3$ by soils was studied by Tacke[1] as early as 1897. The reaction was allowed to proceed for three hours and the CO_2 evolved was expelled by a current of H and determined by absorption in standard $Ba(OH)_2$ solution. It was, however, recognized by the author that more CO_2 was given off when the mixture was heated, but he supposed the amount evolved in the cold to be the true measure of free humic acid in the soil.

Knight modified Tacke's method for determining lime requirement of soil[2]. He boiled the mixture of soil and precipitated $CaCO_3$ for ten minutes in the presence of a neutral salt (it made little difference what neutral salt was used as long as its quantity was not so large as to change materially the boiling point of the solution).

Crowther measured the interaction between $CaCO_3$ and acid soils, in bottles completely filled with suspension, by measuring the total CO_2 liberated as free acid and as bicarbonate.[3] The values thus obtained were found to be close to the amount of alkali required to bring the soil to pH 7. In all the cases noted above, no attempt was made to measure the end point of the reaction, the conditions of experiments being arbitrarily fixed. In the present study, the reaction was allowed to take place for several hours till the evolution of CO_2 practically came to an end.

Ba carbonate was chosen for this purpose. At first it was thought that, since Ba carbonate is insoluble in water, the reaction would only take place in the presence of $BaCl_2$ which will cause the production of HCl that will react with $BaCO_3$ giving CO_2. However, it was soon found that the presence of $BaCl_2$ was not necessary, the reaction proceeded normally in its absence.

[1] Tacke, B. Estimation of free humic acid in Peaty soils. *Chem. Zeit.*, 21, p. 174 (1897).
[2] Knight, H. G. Acidity and acidimetry of soils, IV. Proposed method for determination of lime requirements of soils. *Jour. Ind. Eng. Chem.*, Vol. 12, p. 560 (1920).
[3] Crowther, E. M. The Determination of Lime Requirements. *Agricultural Progress*, Vol. II, (1925).

Fig. 5.

Hydrolysis of ethyl acetate by Clay & Acetic acid
(100 c.c. of Clay suspension contain 2·6 Milli Equivalents of H_1)

Soil Suspension ◯
Acetic acid △

C.C.'s of Clay suspension →
and
Equivalent amounts of Acetic acid →

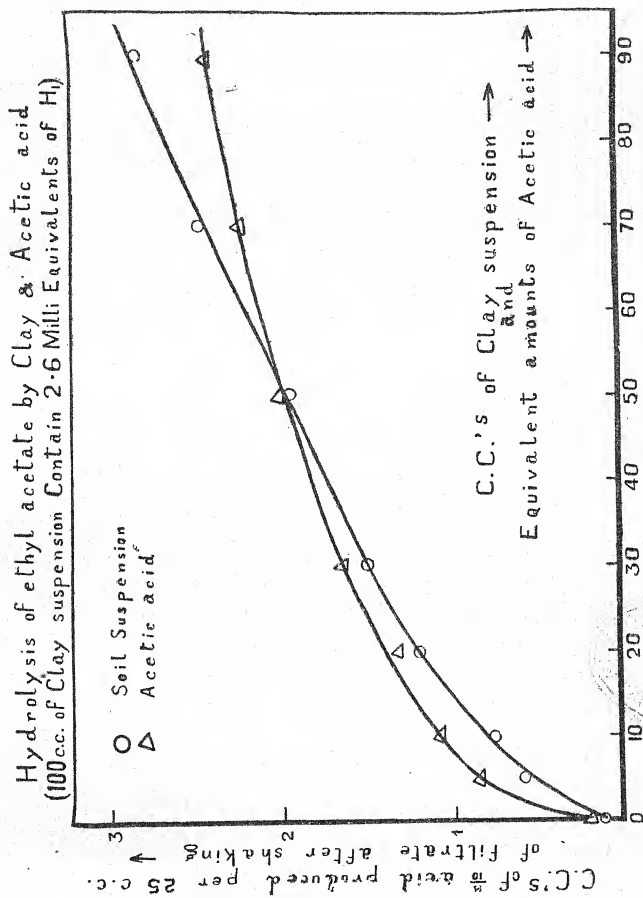C.C.'s of $\frac{N}{10}$ acid produced per 25 c.c.
of filtrate after shaking →

The reaction was found to take place in two stages. Up to one stage it went on in the cold and took about 24 hours for completion and even then perhaps it could not be considered as quite complete. The second stage proceeded more or less rapidly when the mixture was heated. Here the end point is more or less definite. The experiment was conducted in the usual way of determining CO_2 by weighing as well as by titration after absorption in standard Baryta. The end of reaction was determined in one case by weighing the absorption tube and in the other by noting when no precipitate was formed in fresh Baryta in about two hours. CO_2 free air was continuously passed through the apparatus during the course of the reaction.

It was found that the first stage of the reaction (cold) corresponded roughly with the amount of H_1 in the soil as determined by $BaCl_2$ displacement. However, on account of the absence of a definite end point, reaction in the cold was not studied with many soils. On boiling from 6-8 hours the reaction came to an end, and total amount of CO_2 evolved was approximately equivalent to 2 H_1. This was true for a number of soils though perhaps not for all soils as will be seen from Table V. This second equivalent of hydrogen that reacts with carbonate and is not displaced by neutral salts we shall call H_2.

TABLE V.

The relation between H_1 and H_2 for a number of completely unsaturated soils.

Soil									H_1	$H_1 + H_2$		
										$(BaCO_3 + BaCl_2)$	$(BaCO_3)$	$(CaCO_3)$
A. T. 1	.	.	.	.	.	.	.	.	1·4	2·4	2·4	2·8
A. T. 2	.	.	.	.	.	.	.	.	0·8	1·8	1·5	1·94
A. T. 3	.	.	.	.	.	.	.	.	1·0	2·16	2·3	2·23
A. T. 4	.	.	.	.	.	.	.	.	1·7	2·97	3·44	..
A. F. 5	.	.	.	.	.	.	.	.	1·5	2·79	3·0	..
A. T. 6	.	.	.	.	.	.	.	.	0·6	1·59	1·74	..
A. T. 7	.	.	.	.	.	.	.	.	1·1	3·96	2·92	..
A. T. 9	.	.	.	.	.	.	.	.	1·20	2·61	3·28	..

Milli-equivalents per 10 grm. of soil

Table V.—contd.

The relation between H_1 and H_2 for a number of completely unsaturated soils.

Soil	H_1	$H_1 + H_2$ (Milli-equivalents per 10 grm. of soil)		
		$(BaCO_3 + BaCl_2)$	$(BaCO_3)$	$(CaCO_3)$
A. T. 13 .	1·8	..	4·32	..
A. T. 19	1·26	..	2·67	..
A. T. 20	0·5	..	1·26	..
A. T. 26 .	0·62	..	1·56	..
A. T. 28 .	1·30	..	3·33	..
A. T. 29 .	1·10	..	2·28	..

(4) Reaction with a hydroxide and the determination of the hydroxide neutralized.

The state of saturation of the soil (V) is defined by Hissink as the ratio of the amount of absorptively bound bases in the soil (S) to the amount of bases that the soil is capable of binding (T) both values being expressed in equivalents.

Thus $V = \frac{100}{T} S$. The value T-S is the acidic hydrogen, that is, the still " unsaturated hydrogen of the clay and humic acid " and is determined by occasionally shaking during 3 days a known weight of the soil with increasing concentrations of baryta. At the end of four days the clear liquid is pipetted off and titrated to phenolphthalein with acid, when the amount of base removed by the soil is plotted against the amount of base remaining in solution ; a curve similar to the one shown in Fig. 6 is obtained. The straight part of the curve (the baryta branch) is produced till it meets the vertical axis. The point of intersection is taken to be the point of neutralization or " saturation point." The first, *i.e.*, the curved portion of the curve is supposed to be due to hydrolysis by analogy to the neutralization of a soluble weak acid with a strong base. (Saint in *Trans. Int. Soc. 2nd Commission*, Vol. A, p. 135).

Such curves were obtained for a number of soils and a typical one for Akola soil is shown in Fig. 6.
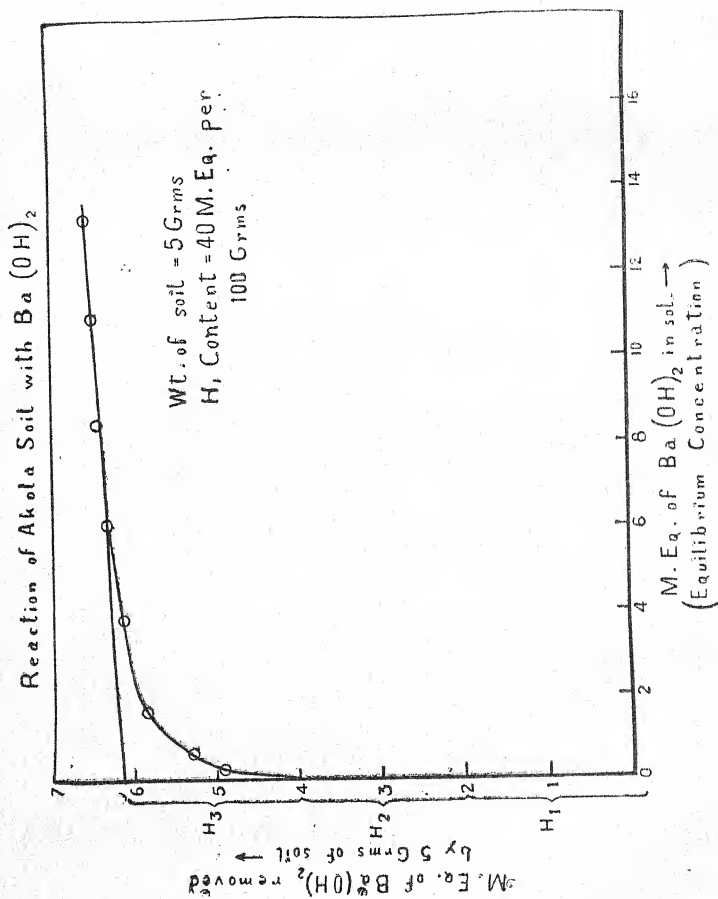
Fig. 6.

Reaction of Akola Soil with Ba(OH)$_2$

Wt. of soil = 5 Grms
H, Content = 40 M.Eq. per
100 Grms

M.Eq. of Ba(OH)$_2$ in sol.
(Equilibrium Concentration)

M.Eq. of Ba(OH)$_2$ removed
by 5 Grms of soil

Five grams of the air-dry soils were in each case shaken with increasing concentrations of baryta. The curves can be extrapolated back in two directions, namely, from the straight portion or from the curved part the latter can only be taken as approximate. The values of the acid displaced by $BaCl_2$ (H_1 content referred to before) as well as the CO_2 evolved by heating with $BaCO_3$ are indicated on the curve (all quantities expressed as milli-equivalents per 5 grams of the soil).

It will be seen that if the straight portion of the curve is extrapolated as according to Hissink or Saint we get a very high value which is three times the amount of acid that can be displaced by neutral salt reaction (H_1). This extra amount of hydrogen over and above H_1 and H_2 we shall call H_3. If, on the other hand, the curved portion is extrapolated, we get a figure which is almost equivalent to the amount of CO_2 evolved by $BaCO_3$ treatment and likewise is twice the equivalent amount of H_1 (Table VII). Thus we see that a completely unsaturated soil contains 3 grm. equivalents of hydrogen ($H_1 + H_2 + H_3$) : H_1 can be displaced by the action of a neutral salt, H_2 by boiling with Ba carbonate and H_3 by the action of $Ba(OH)_2$

(5) REACTION WITH A SULPHIDE AND DETERMINATION OF H_2S EVOLVED.

This reaction has not been studied strictly quantitatively. However, the field outfit of Truog based on the same principle was used on a number of soils and the order of acidity denoted was always the same as that determined by $BaCl_2$ replacement.

Truog [1] employs a mixture of $BaCl_2$ and ZnS which is heated with the soil suspension and the amount of H_2S liberated is indicated by the depth of colour produced on a lead acetate test paper. Parker and Tidmore [2] have suggested a modification of Truog test, which consists in absorbing the H_2S evolved in dilute ammonia. An excess of standard iodine solution is then added, the solution acidified and the excess of iodine titrated with standard sodium thiosulphate of the same normality. They found that increasing the quantity of soil used in making the test increases the amount of H_2S evolved but not proportional to it. Increasing the quantity of reagent above 1·1 gram does not increase and may decrease the amount of H_2S evolved. The reaction is allowed to take place only for 3 minutes, and other defined conditions must be adhered to rigidly.

It seems the beauty of the Truog test lies in its simplicity, and utility as a field method. The writer made several fruitless attempts to study the reaction quantitatively : the main difficulty lies in the fact that a mixture of $BaCl_2$ and ZnS slowly gives H_2S when boiled even without any soil, which itself is a complicated reaction influenced by several factors. However, it was shown quite definitely that the rapid production of H_2S only took place in the presence of soils having free H_1.

[1] Truog, E. A new test for soil acidity. *Wis. Agri. Expt. Sta. Bull.* No. 249 (1915) ; and testing soils for acidity, *Bull* 312, (1920 ; revised 1924).
[2] Parker, F. W., and Tidmore, J. W. A modification of the Truog soil acidity test. *Soil Sci.*, Vol. 16, p. 75 (1923).

(6) Reaction with ammonia.

If excess of $\frac{N}{10}$ ammonia is added to soil and the mixture boiled for two hours or left in the oven to dry (at 100-105°C), then the amount of ammonia that is still retained by the soil is nearly equivalent to H_1 (Table VI). This affords an easy method of finding H_1 in a fully unsaturated soil. The method, however, could not be used for finding the degree of unsaturation of partly unsaturated soils as the nature of the exchangeable ion already present modified the retention of ammonia under the above set of conditions. What happens is that a part of the base is replaced by ammonia and the latter is retained to a larger extent than the amount equivalent to the degree of unsaturation of the soil (with respect to H_1). This point will be discussed more fully in Part IV of these studies.[1]

The methods of estimating H_1 are not very exact and the agreement in the two sets of values shown in Table VI is really better than what it appears, considering the nature of a substance like soil. On the whole, boiling for two hours gives values that show a better agreement with H_1 than oven drying.

Table VI.

The relation between H_1 content and ammonia absorption at 100°C.

Soil									H_1	Ammonia Oven drying	Boiling 2 hours
A. T. 1	.	.	.	.	.	.	.	.	1·4	0·99	1·07
A. T. 2	.	.	.	.	.	.	.	.	0·8	0·73	0·65
A. T. 3	.	.	.	.	..	.	.	.	1·0	0·92	0·87
A. T. 4	.	.	.	.	.	.	.	.	1·7	1·12	1·32
A. T. 5	.	.	.	.	.	.	.	.	1·5	1·20	1·48
A. T. 6	.	.	.	.	.	.	.	.	0·6	0·49	0·65
A. T. 7	.	.	.	.	.	.	.	.	1·1	0·8	1·23
A. T. 9	.	.	.	.	.	.	.	.	1·2	1·6	1·67
A. T. 13	.	.	.	.	.	.	.	.	1·8	1·31	1·76
A. T. 19	.	.	.	.	.	.	.	.	1·2	0·95	1·21
A. T. 20	.	.	.	.	.	.	.	.	0·5	0·26	0·39
A. T. 26	.	.	.	.	.	.	.	.	0·62	0·46	0·76
A. T. 28	.	.	.	.	.	.	.	.	1·3	1·78	1·27
A. T. 29	.	.	.	.	.	.	.	.	1·1	0·77	..
Clay fractions (·001 mm.) from various soils.—											
F. 1	.	.	.	.	.	.	.	.	2·3	2·3	..
F. 2	.	.	.	.	.	.	.	.	3·1	3·5	..
F. 3	.	.	.	.	.	.	.	.	3·2	2·5	..
F. 4	.	.	.	.	.	.	.	.	4·1	3·6	..
F. 5	.	.	.	.	.	.	.	.	0·5	0·7	..
F. 6	.	.	.	.	.	.	.	.	2·6	2·9	..

Milli-equivalents per 10 grm. of soil

[1] Puri, A. N.   Studies in Soil Colloids, Part IV, Methods of estimating soil colloids.   *Mem. Dept. Agri. India, Chem. Ser.*, Vol. XI, No. 7.

(7) REACTION WITH AMINES.

Di-ethyl amine was employed for this purpose. As in the case of ammonia absorption about 40 c.c. of $\frac{N}{10}$ solution of the amine were left with 5 grams of soil in the oven (105-110°C) overnight, next day amine was determined by steam distillation in the presence of excess of lime. The results were similar to that of ammonia. That is, the amine reacted with H_1 only under the above set of conditions.

All the reactions detailed above were examined with half a dozen soils and the results given in Table VII show their interrelationship. The agreement is as good as could be expected from a mixture of various alumino-silicates constituting soils. There are exceptions for which no explanation can be found. Red soils as a rule gave a very low value for H_1 as compared to H_2. The subject needs further study with various types of soils. However, there can be no question as to the three stages of reaction designated as H_1, H_2 and H_3 : though their chemical equivalence may be doubted. Nor is it expected that all soils will show such a simple relationship ; *but the fact that a number of soils could be picked up at random which undoubtedly do show such regularities is highly significant.*

TABLE VII.

Relation between H_1, H_2 and H_3 in various soils completely unsaturated.

Soils	Clay (·002 mm.) per cent.	Hygros-copicity (50 per cent. humidity)	Heat of wetting (calories per grm. of soil)
(1) Bangalore (Red soil)	18·4	0·68	1·18
(2) Godavari	37·2	4·04	3·70
(3) Shillong (Red soil)	36·2	3·42	3·75
(4) Koilpatti	49·8	10·05	9·00
(5) Akola	60·6	11·31	9·50
(6) Dharwar	60·5	10·67	8·75

Reactions characteristic of H_1 (Milli-equivalents per 10 grams of soil).

Soil No.	Displacement with $BaCl_2$	Titration to B. T. blue	Ammonia boiling	Ammonia oven drying	Ethyl amine	Mean H_1
1	0·28	0·65	0·4	··	0·32	0·41
2	1·17	1·70	1·60	1·58	1·58	1·53
3	1·33	1·50	1·98	1·67	1·38	1·57
4	4·12	4·22	3·50	4·40	4·52	4·15
5	4·16	5·10	4·46	4·62	4·46	4·36
6	3·39	3·60	2·98	3·92	3·54	3·49

Reactions characteristic of H_2 and H_3 (Milli-equivalents per 10 grams of soil).

Soil No.	$(H_1+H_2)=2H_1$			$(H_1+H_2+H_3)=\frac{3}{2}(H_1+H_2)$	
	CO_2 evolution with $BaCO_3$	$Ba(OH)_2$ Extrapolation	Mean H_1+H_2	$Ba(OH)_2$ found	(T Hissink) calculated (from H_1+H_2)
1	1·10	1·40	1·25	1·96	1·87
2	3·67	3·70	3·68	5·90	5·52
3	5·50	5·50	5·50	7·80	8·25
4	7·58	7·00	7·29	9·60	10·93
5	8·26	8·00	8·13	12·00	12·19
6	6·82	7·00	6·91	9·60	10·36

(8) HYDROGEN ION CONCENTRATION OF THE SUSPENSION.

The hydrogen ion concentration of a number of soils (Table VIII) was determined with special reference to the degree of unsaturation in each case. The results given in Table IX leave no doubt that the pH value of a soil suspension gives us no indication of its degree of unsaturation or the amount of surface active hydrogen ions. pH value of a soil suspension represents a state of dynamic equilibrium which could only be defined with reference to a particular set of conditions and it leaves out of account the most fundamental property, *i.e.*, the state of saturation of the soil or the amount of surface active hydrogen ions.

The H electrode measures the hydrogen ion concentration of the solution only and is very little affected by the surface active hydrogen ions on the soil particles. It is also possible that H concentration of soil in KCl solution might give us a better idea of the state of saturation, as the value thus obtained will be directly related to the degree of saturation of the soil.

TABLE VIII.

Soils used for pH determination.
Series I.

Completely desaturated soils by exhaustive treatment with $\frac{N}{20}$ HCl and subsequent washing with water.

(1) Akola H soil.
(2) Soil A. T. 18 Michigan loam.
(3) Rendzina H soil.
(4) Hoos Fallow (Rothamsted) H soil.
(5) Montgomery H soil.
(6) Humus H soil.
(7) Jokak H soil.

Series II.

Series containing increasing amounts of Ba in the exchange complex obtained by treating Akola H soil with increasing amounts of $BaCl_2$ and washing (the same as used for sugar inversion).

TABLE IX.

Showing the pH value of various soils (Acid soils shaken for 2 hours, Ba soils for 6 hours) E. M. F. found out in complete absence of KCl in the soil solution vessel. Ratio of soil and water is 1 : 5.

Soil										H$_1$ content Milli-equivalent per 10 grm. of soil	pH
Akola H soil	.	.	.	.	.	.	.	.	.	45·3	4·6
Michigan loam	.	.	.	.	.	.	.	.	.	17·10	4·31
Rendzina H soil	.	.	.	.	.	.	.	.	.	16·39	4·12
Hoos fallow H soil	.	.	.	.	.	.	.	.	.	15·30	4·55
Montgomery H soil	.	.	.	.	.	.	.	.	.	9·40	5·54
Humus H soil	.	.	.	.	.	.	.	.	.	13·0	4·80
Gokak H soil	.	.	.	.	.	.	.	.	.	14·30	4·88
Akola Ba Soil series.											
No. 1	.	.	.	.	.	.	.	.	.	28·4	4·51
,, 2	.	.	.	.	.	.	.	.	.	15·8	4·52
,, 3	.	.	.	.	.	.	.	.	.	11·8	4·55
,, 4	.	.	.	.	.	.	.	.	.	8·1	4·57
,, 5	.	.	.	.	.	.	.	.	.	6·8	4·645
,, 6	.	.	.	.	.	.	.	.	.	5·2	4·70
,, 7	.	.	.	.	.	.	.	.	.	0	6·92

In this connection might be mentioned the results of Truog [1] and Pierre[2], both of whom found that, after washing away thoroughly the soluble salts and acids, the effect of soil water ratio on the hydrogen ion concentration of the suspension is *nil*.

Joffe and Mclean[3] also concluded from their studies that the measurement of the H ion concentration in a water extract of soil add very little to our knowledge of the state of saturation or unsaturation; they give only an idea of the free acid present. We may thus suppose with Michaelis[4] that "Colloid acids, as common acids suspended in pure water, increase the quantity of H ions, but they do not increase the concentration of H ions at any place within the interior of the soil. They only bring about an accumulation of H ions round the Micellae." We may also recall several attempts at finding a relation between the hydrogen ion concentration in soils and their lime requirements. This point has been dealt with exhaustively by Johnson [5] who arrived at the conclusion that in soils as a whole the apparent quantity of acid or the lime requirement has no relationship to the intensity or

[1] Truog, E. The Colloid Chemistry of Soils. *Colloid Symposium Monograph*, Vol. II.
[2] Pierre, W. H. The hydrogen ion concentration of soils as affected by CO$_2$ and the soil water ratio and the nature of soil acidity as revealed by these studies. *Soil Sci.*, Vol. 20, p. 285, (1925).
[3] Joffe, J. S., and Mclean, H. C. Colloidal Behaviour of Soils and Soil Fertility, II. The Soil Complex capable of Base exchange and Soil Acidity. *Soil Sci.*, Vol. 21, p. 181 (1926).
[4] Michaelis, L. General Principles of the effect of ions in Colloids. *Colloid Symposium Monograph*, Vol. II, (1925).
[5] Johnson, H. W. The relation of hydrogen ion concentration in soils to their lime requirement. *Soil Sci.*, Vol. 13, p. 7, (1922).

strength of acids present, or the pH value. This substantiates the inferred conclusion of Joffe [1] that sands and clays vary in relation between these two factors. However for soils of similar origin the two factors probably bear a close relationship to each other. This explains the conclusions of Blair and Prince [2] who found that there was a definite relation between the lime requirement and hydrogen ion concentration of soils from the series of plots at the New Jersey Station.

(9) REACTION WITH NEUTRAL SALTS.

This is the most widely known reaction of acid soils and probably the earliest to attract the attention of soil scientists. Several methods of finding lime requirements of acid soils are based on neutral salt decomposition with the production of free soluble acids which can be determined easily. Among these may be mentioned Hopkin's [3], Daikuhara's [4] and Gedroiz's method [5] who use KNO_3, KCl, and $BaCl_2$ solution respectively to liberate free acids.

As mentioned before, the reaction between a neutral salt and an acidoid represents a state of equilibrium depending upon the concentration of the reacting bodies. However, if the concentration of one of the reacting bodies be increased considerably, the reaction could be pushed in one direction and we might be able to get approximately to the end point.

In order to see what percentage of the total H_1 content of a soil could be replaced by a single treatment of a neutral salt ; 5 grm. portions of a soil were shaken for 2 hours with 100 c.c. solutions of varying concentrations (from $\frac{N}{50}$ to 4 N) of the following salts :—

$LiCl$, NH_3Cl, KCl, $MgCl_2$, $SrCl_2$, $CaCl_2$, $BaCl_2$, NH_4NO_3, $NaNO_3$, KNO_3, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Ba(NO_3)_2$, $(NH_4)_2SO_4$, Na_2SO_4, K_2SO_4.
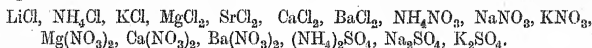
After two hours the suspension was filtered and 50 c. c. of the filtrate titrated against standard alkali to phenolphthalein (in the case of Mg and ammonium salts the titrations done in the presence of Brom Thymol Blue). The results shown in Fig. 7 indicate that although there are individual variations in different salts at lower concentrations, those differences tend to become small in higher concentrations and almost disappear in the neighbourhood of about 2 N. Nitrates on the whole show a slightly lower value than the chlorides or sulphates. The maximum value represents about 90-95 per cent. of the total H_1 content determined by exhaustive treatment with $BaCl_2$ solution.

[1] Joffe, J. S. Hydrogen ion concentration measurements of soils in connection with their lime requirements. *Soil Sci.*, Vol. 9, p. 261, (1920).

[2] Blair, A. W., and Prince, A. L. *Soil Sci.*, Vol. 9, p. 253, (1920).

[3] Hopkins, Soil Fertility and Permanent Agriculture. *U. S. Dept. Agri. Bur. of Chem. Bull.* 73, (1903).

[4] Daikuhara, G. Ueber saur mineral Boden. *Bull. Jap. Imp. Centr. Agri. Exp. Sta.*, 2, p. 1, (1914).

[5] Gedroiz, K. K. *Zhur. Opit. Agron.*, 22, p. 3, (1924).

Fig. 7.

Reaction of unsaturated Soil with Neutral salts.
(56rms of Soil shaken with 100 c.c.)
of solution

(10) HEAT OF NEUTRALIZATION.

It is well known that for most acids the heat of neutralization with bases in equivalent concentrations is approximately the same, since in all cases it represents a union between H and OH ions.

In order to study the thermal effect on formation of the salts of acidoid, particularly with reference to H_1, H_2 and H_3 three clay suspensions were used. The clays ($>·001$ mm. diam.) were separated after acid treatment from three different soils.

These were :—

    1. Dharwar black cotton soil—

        H_1 content$=6·6$ milli-equivalents per 10 grm. of dry clay.

    2. South African red soil—

        H_1 content$=2·38$ milli-equivalents per 10 grm. of dry clay.

    3. Pusa soil—

        H_1 content$=1·34$ milli-equivalents per 10 grm. of dry clay.

The heats of neutralization of one gram atom of H_1, H_2 and H_3 in each case is given in Table X. The results for the heats of neutralization of one gram molecule of acetic acid determined under the same set of conditions are included for comparison.

TABLE X.

Heat of neutralization of acidoid and acetic acid with various bases.

	Portion of acidoid neutralized	HEAT OF NEUTRALIZATION (CALORIES) PER ONE GRAM MOLECULE OF ALKALI			
		Clays			Acetic acid
		(1)	(2)	(3)	
NH_4OH	H_1	6041	6060	5353	11770
	H_2	1681	1428	1122	
	H_3	..	309	927	
$LiOH$	H_1	5507	5454	4545	12390
	H_2	3643	3469	2366	
	H_3	1166	2060	736	
$NaOH$	H_1	5487	5857	5151	12130
	H_2	2879	3774	1937	
	H_3	543	1339	721	
KOH	H_1	5983	6666	6661	11770
	H_2	3720	3876	3366	
	H_3	562	1133	1545	

A glance at Table X will reveal some interesting facts ; for instance it appears that the heat of neutralization of H_1 in the 3 clays is approximately the same for the 4 bases studied and also this is of the same order as the heat of neutralization of the first gram atom of hydrogen for silicic acid ($=4316$ C).

The neutralization of H_2 is accompanied by a smaller heat evolution which depends on the nature of the base and perhaps the nature of the soil. Ammonia especially gives a very small heat of neutralization of H_2. The neutralization of H_3 with an equivalent amount of alkali takes place with a much smaller heat production than H_1 and H_2 which is also dependent on the nature of alkali and perhaps the nature of the soil.

The calculations are based on the exact determination of H_1 and at least in the case of clay No. 3 this is so small that a considerable error might be introduced in the final computation of the results. In spite of the errors involved in such measurements, there is a conspicuous uniformity in the results which brings the acidoid into line with other soluble acids.

It might be mentioned that after the addition of 3 gram molecules of alkali to neutralise H_1, H_2, H_3, an appreciable though extremely small rise of temperature takes place on the addition of further amounts of alkali. This is not altogether unexpected considering that the salts of H_3 are so easily hydrolised and that the neutralisation of H_3 takes place only in the presence of large excess of alkali.

THEORY OF SOIL ACIDITY AND ITS PRACTICAL IMPORTANCE.

A number of reactions have been studied and discussed, it now remains to be seen if we can build up a comprehensive theory to explain all the phenomena noted above.

The outstanding facts are :—

    (1) The soil can be completely desaturated by exhaustive treatment with $\frac{N}{20}$ HCl which has only slight dissolving action on the exchange complex. Such a soil contains a definite quantity of surface active hydrogen ions giving it the character of an " acidoid."

    (2) The " acidoid " thus produced shows reactions characteristic of true acids. These reactions, however, take place in three distinct stages in which one, two or three equivalents of H can take part, *i.e.*, H_1, H_2 and H_3. H_1 is replaced by exhaustive treatment with a neutral salt, when this is replaced the suspension is neutral (pH7). H_1 is the only part that can invert sucrose, hydrolyse Ethyl acetate, decompose ZnS, show " exchange acidity," or retain ammonia or amines at 100—105°C.

The statement that the final pH of the suspension is$=7$ when it is treated exhaustively with a neutral salt needs some amplification as apparently it is not corro-

borated by the experimental results of other workers, like Kelley and Brown [1] and Parker.[2] The explanation of this apparent discrepancy is simple and is comprised in the following general statement : *that the soil tends to impart its own pH to the solution with which it comes in contact and vice versa.* Therefore if a soil is leached with a solution, then the former must ultimately acquire the same pH as the latter. If it is washed with a neutral salt having a pH value less than 7, then obviously the soil can never acquire a pH of 7, it can only do so if the neutral salt is first brought to pH 7, before leaching the soil with it. The procedure followed by the writer in this connection was to add about 200 c. c. of N. $BaCl_2$ to 10 grams of soil, the filtrate after shaking the mixture for a few minutes was neutralized with $Ba(OH)_2$ using Brom Thymol Blue as indicator, and poured back on to the soil, the filtrate again neutralized as before and the process repeated till no more acid was liberated from the soil. Later on this procedure was simplified by adding excess of $BaCl_2$ to the soil suspension along with about 10 drops of Brom Thymol Blue (1 per cent. sol). The acid liberated titrated in the presence of the soil which settles down quickly leaving a clear supernatent liquid in which the colour change of the indicator can be followed.

When H_1 has been replaced in a soil, another equivalent of hydrogen (H_2) can take part in reaction with carbonates with the evolution of CO_2. After reaction with carbonates, it still contains another equivalent of hydrogen H_3 that can neutralize alkalies. This last compound, however, is easily hydrolysed, so the end point can only be determined by having a large excess of alkali and extrapolating to zero concentration of alkali in solution (Hissink).

The above experimental facts can be best interpreted by supposing that the insoluble colloidal acid (" acidoid ") in a fully unsaturated soil is a tribasic acid which undergoes the following reactions :—

1. *Di-hydrogen acid salt formation.* (Neutralization of H_1)—
    (a) Takes place by treating exhaustively with a neutral salt (pH7).
    (b) Treating with ammonia solution and driving away the excess by heating at 100-105°C overnight.
    (c) Neutralisation with alkali to pH7.

2. *Monohydrogen acid salt formation* (neutralization of H_1 and H_2)—
    (a) Takes place by heating the suspension with Ca or Ba carbonate in a current of air.

3. *Normal salt formation.* (Neutralization of H_1, H_2, and H_3).

Takes place in the presence of large excess of alkali and the neutralization point can be determined by treating with increasing concentrations of $Ba(OH)_2$ solution, and extrapolating the straight portion of the curve (Hissink).

[1] Kelley, W. P., and Brown, S. M. Ion exchange in relation to soil acidity. *Soil Sci.*, **21**, p. 289, (1926).
[2] Parker, F. W. Methods for the determination of the amount and acidity of exchangeable hydrogen in soils. *Proc. First Int. Cong. Soil Sci., Washington*, Vol. II, p. 164 (1928).

If this theory is correct, then we should expect breaks in the titration curves corresponding to the various stages of reaction.

Some evidence on this point has already been brought forth by Bradfield (*J. A. C. S.*, Vol. XIV, 1923, p. 2669) who says : " The curves (titration) obtained by the hydrogen-electrodes are also of the type usually obtained with very weak acids. There are no sharp breaks, but the slope changes gradually, showing the least buffer action between pH 9·5 and 7.   At the latter point there is a slight flattening of the curve with all clays, studied.   It seems to be as pronounced as that observed in the titration of certain weak crystalloidal acids such as phthalic acid and would seem to be evidence that at least a dibasic acid is involved."

One such curve obtained in the case of a clay suspension by the writer is shown in Fig. 8, but a number of such curves will have to be determined for different soils before it could be established whether the change of slope occurs at the same pH value for all soil suspensions.   In this case it occurs at pH 6·5 and the point corresponds with the neutralization of H_1, the other change occurs at pH 10-11 and corresponds to the neutralization of both H_1 and H_2.   Bradfield's curves are similar to the one shown.

C.C.'s of $\frac{N}{10}$ Ba(OH)$_2$ added to 50 c.c. of the suspension →
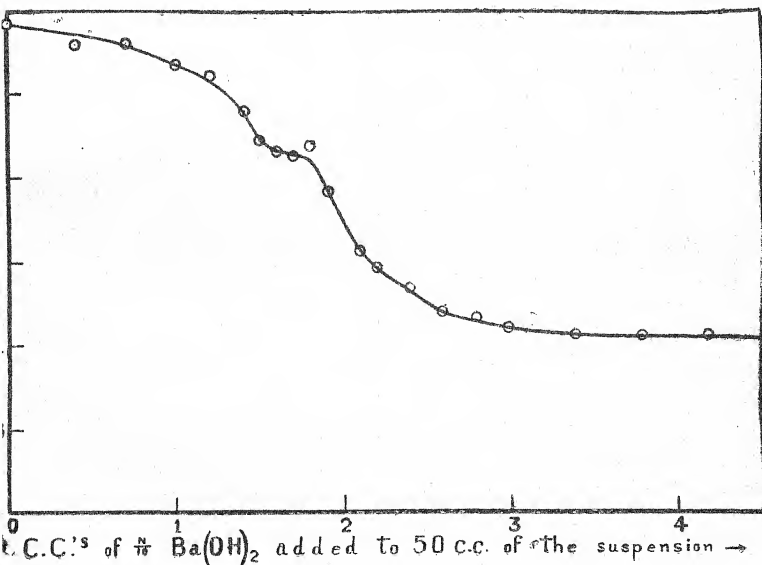
Fig. 8.

In the light of the above theory, we can find a more rigid definition for acid and alkali soils.

Acid soils are those in which a part, or the whole of H_1 is free (the latter, being an extreme case, is seldom found in nature).

Alkali soils are those in which the whole of H_1 and a part or the whole of $_4H_2$ has been replaced or neutralized.

Different forms of soil acidity designated as " exchange " " active " and " hydrolytic ", etc., lose a good deal of their former significance when viewed in the light of the above hypothesis ; for instance, active or exchange acidity refers to H_1 only and hydrolytic acidity takes into account a part of H_2 also.

Attention may again be directed to a single value factor of great importance, a characteristic constant of great value for all studies of soil colloidal matter, $i.e.$, the maximum amount of surface active hydrogen that a soil can develop ; (expressed as milli-equivalents per 100 grams of soil).

This factor has already been referred to by Hissink as " Saturation capacity " or " Absorbing capacity " and by Gedroiz as the " Volume of adsorption," though Hissink's saturation capacity may be equivalent to three times Gedroiz's volume of adsorption. It has also been suggested that this factor should be employed for the genetic classification of soils. A view which will meet with general approval.

A critical review of the methods of determining " Saturation capacity " of soils will be made in part V of this series. As has been hinted above, a good deal of confusion exists in soil literature as to the precise meaning to be attached to this factor, and it is probable that the methods of determining it will meet with the same fate as the methods of finding lime requirements of acid soils : all purporting to measure the same thing yet giving widely different results.

Incidentally the ammonia absorption experiments discussed above point strongly to the fallacy of experiments designed to measure the colloidal content of soils by ammonia absorption, or by the adsorption of basic dyes. Some correlation there must be in similar soils where the state of saturation of the soil colloids is the same, but the method cannot measure the colloidal content of a soil.

If the exchange complex is an alumino silicate and if it is found to be tribasic, we may perhaps, with some reason, express the reaction between aluminum hydroxide and meta silicic acid as follows :—

$$Al_2 (OH)_6 + 2H_2 SiO_3$$
$$\longrightarrow \{Al (OH)_2\}_2 Si_2 O_5 + 3 H_2 O$$

or

$$Al \diagdown\begin{array}{l} OH \\ SiO_4 \equiv Al \\ SiO_4 \equiv H_3 \end{array}$$

" Acidoid."
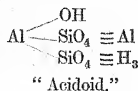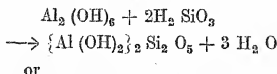
An attempt will be made to prepare this compound and study its properties. The above constitution might explain why Al is invariably associated with soil acidity

and also why Al always keeps coming in solution when the soil is washed with dilute acids. The maximum value on the curve No. 1 might also be partly conceivable. But it is too early to make any guesses.

REVIEW OF THE METHODS OF FINDING LIME REQUIREMENTS OF ACID SOILS, IN THE LIGHT OF THE TRIBASIC ACIDOID THEORY, AND AN ACCOUNT OF FIELD EXPERIMENTS ON LIMING.

It is not intended to make a critical examination of the existing methods of finding lime requirements ; a few remarks, however, will not be out of place.

Lime requirement methods may be grouped under the following general heads :—

    (1) *Reaction with neutral salts* such as KNO_3 (Hopkin) ; KCl (Diakuhara also Kappen) ; $BaCl_2$ (Gedroiz). The last named has suggested exhaustive treatment, others single treatment.

    (2) *Reaction with hydrolysed salt* such as sodium or potassium acetate (Loew) calcium acetate (Jones and also Kappen).

    (3) *Reaction with alkali* such as $Ca(OH)_2$ (Veitch) ; $Ca(HCO_3)_2$ (Hutchinson and Maclennen), $Ba(OH)_2$ (Hissink, also Lyon and Bizzel, also Truog). Neutralization to pH7.

    (4) Reaction with ZnS_7 and measurement of H_2S evolved. First suggested by Truog and later modified by Parker and Tidmore.

    (5) Reaction with a carbonate and determination of CO_2 evolved ; in the cold (Tacke) ; or on heating (Knight).

It will be clear from the results of the experiments with completely unsaturated soils that the above methods, though intended to give the same result, will furnish widely differing values for lime requirement. No. 1 method will give about 75 to 90 per cent. of H_1, except the method of Gedroiz which will give as near a value to H_1 as is possible by treatment with a neutral salt.

No. 2 method will give the value for H_1 and a greater or lesser portion of H_2 depending upon the experimental conditions.

No. 3 method will give the whole of H_1 and H_2 and perhaps a part of H_3 also depending upon the conditions of experiment ; except neutralization to pH_7 which will give the value for H_1 only.

No. 4 method is only qualitative. Though the quantity of H_2S evolved is proportional to H_1, it is not possible to determine H_1 from the values obtained by these methods.

No. 5 method will give the value for H_1 and a part of H_2 depending on the time of reaction.

It is difficult to choose from the above methods until the relative toxicity of H_1 and H_2 has been established from pot culture experiments and field trials. Theoretically the lime hunger of a soil could be fully satisfied only when it is neutralized

C

up to the H_3 stage (Hissink's method of finding the state of saturation of a soil). From the practical point of view, however, it is quite immaterial whether the whole of the hydrogen is neutralized or only a part, as long as we can get the maximum yield with the minimum dose of lime. Plant must be the ultimate criterion to show what proportion of H_1 can be tolerated by it, and what proportion of H_2 if neutralized would be beneficial.

Since H_1 was the only part that could invert cane sugar, hydrolyse Ethyl acetate, decompose zinc sulphide quickly, show " exchange acidity " or retain ammonia and amines at 100—105°C, *i.e.*, in general show reactions characteristic of true acids ; it was believed that the toxicity of acid soils would be mainly due to H_1. Therefore in the liming experiments described here, the basis of lime requirement was the H_1 limit determined by neutralization of the soil suspension to Brom Thymol Blue in the presence of excess of $CaSO_4$.

The red lateritic soil of Dacca which is typical of a large part of Bengal was chosen for these experiments. The soil is highly acidic and possesses an open texture through which water percolates rapidly though the clay content is fairly high (38 per cent).

A two acre plot of land was divided into 4 parts and manured as follows :—

    A. No manure.
    B. Mustard cake to supply 40 lb. of nitrogen per acre.
    C. Ammonium sulphate to supply 40 lb. of nitrogen per acre, and bone to
        supply 40 lb. of phosphorous per acre.
    D. Farmyard manure—200 maunds per acre.

Each of these four plots was divided into 12 sub-plots, six of which formed the *a* series and the other six, *b* series, *a's* being duplicates of *b's* (Fig. 9).

About half a dozen soil samples were taken from each of these sub-plots, mixed and air dried and then tested for H_1 limit. Lime was then added according to the following scheme :—

    1. Control, (no lime).
    2. $\frac{H_1}{3}$ (average dose 11·7 md. per acre).
    3. $\frac{2H_1}{3}$ (average dose 23·4 md. per acre).
    4. H_1 (average dose 35·1 md. per acre).
    5. $\frac{3H_1}{2}$ (average dose 52·6 md. per acre).
    6. 2_{ir_1} (average dose 70·2 md. per acre).

The sub-plots, therefore, did not actually receive a regularly increasing dose of lime, but this was regulated according to the H_1 limit in each sub-plot. The unevenness in the entire field was thus eliminated to a certain extent, and the sub-plots received increasing doses of lime on the fundamental basis of H_1. The actual amounts of lime in maunds per acre applied to each sub-plot is stated in Fig. 9.

AMAR NATH PURI 33

b Series   *a Series*   *a Series*   *b Series*

Am. Sulphate + Bone

77·9 | 62·3   **Farmyard manure**   69·7 | 69·7

51·8 | 51·8   46·2 | 51·8

34·5 | 33·1   35·7 | 34·5

24·9 | 20·5   25·7 | 21·7

Control (No lime) 14·6 | Control (No lime) 10·5 | Control (No lime) 14·3 | Control (No lime) 11·7

←·12·5 ft.→

69·6 | 74·9   65·9 | 72·0

73 | 55·9   45·1 | 45·1

No manure 48·8 | 38·3   **Mustard Cake** 35·1 | 35·1

25·2 | 22·5   21·1 | 20·5

Control (No lime) 12 mds. | Control (No lime) 11·1 mds. | Control (No lime) 9·9 mds. | Control (No lime) 9·9 mds.

Road →

b Series   *a Series*   *a Series*   *b Series*

Fig. 9.

c

A crop of *juar* (*Sorghum vulgare*) was put in on the 20th September and harvested on the 30th November 1928. The yield from various plots is recorded in Table XI and graphically shown in Fig. 10. Due to unevenness of the *b* sub-plots and the direction of the natural drains, the results are not so uniform as in *a* sub-plots. In the graph, only the results of *a* sub-plots are plotted.

TABLE XI.

Showing the yield in lb. from various sub-plots in liming experiments (the crop was harvested and weighed immediately).

Sub-plot									A (No manure)	B (Mustard cake)	C (Am. sulph. + Bone)	D (Farmyard)
0	a_1	.	.	.	.	.	.	.	7	42	2·5	32
	b_1	.	.	.	.	.	.	.	17	76	24·6	7
$\frac{H_1}{3}$	a_2	.	.	.	.	.	.	.	18	263·5	113	215·5
	b_2	.	.	.	.	.	.	.	25	214	88	168
$\frac{2 H_1}{3}$	a_3	.	.	.	.	.	.	.	45	199	185	300
	b_3	.	.	.	.	.	.	.	50	233	98	279
H_1	a_4	.	.	.	.	.	.	.	46·5	212	197·5	349·5
	b_4	.	.	.	.	.	.	.	11	209·5	156	279·5
$\frac{3 H_1}{2}$	a_5	.	.	.	.	.	.	.	27	199·5	186	413
	b_5	.	.	.	.	.	.	.	37·5	222	96	397
$2 H_1$	a_6	.	.	.	.	.	.	.	4	77	166	319·6
	b_6	.	.	.	.	.	.	.	32	78	116	404·5

DISCUSSION OF RESULTS.

The results shown in Fig. 10 bring out the following interesting facts :—

    (1) Manuring without lime or liming without manure does not improve these soils much. The ultimate solution will lie in finding a judicial combination of the two, *i.e.*, liming and manuring.
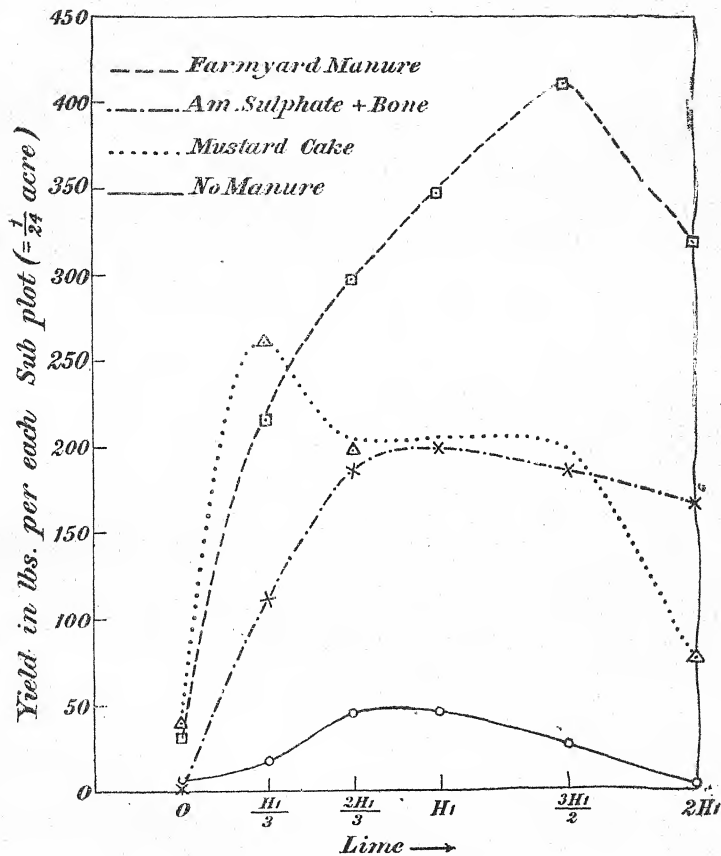
Fig. 10.

(2) The optimum dose of lime is different in the presence of different manures :
For instance, with mustard cake, the optimum is reached with a very much smaller dose of lime than in the case of ammonium sulphate or farmyard manure. It is, however, apparent that the larger dose of lime required in the case of farmyard manure results in an increased crop yield whereas with ammonium sulphate we require about three times the amount of lime to get the same yield as mustard cake.

(3) After the maximum value for the crop yield is reached in the case of all the manures and even no manure, there is a distinct tendency for the crop to fall in yield. This is very much more marked in the case of mustard cake than any other manure.

A possible explanation of the peculiar behaviour of mustard cake is furnished by the fact that the pH value of an acid soil increases when it is shaken with mustard cake, showing thereby that less lime will be required to neutralize (bring it to pH7) an acid soil in the presence of mustard cake. This probably explains the fact that the optimum dose of lime for plots supplied with mustard cake is very much lower than in other plots. It is also shown from nitrification studies of acid soils conducted in the Bacteriological Section of this Institute that the increasing doses of lime added to the soil result in an increased production of nitrites in the presence of mustard cake. These nitrites are decomposed at a later stage, but this disappearance of nitrites takes place after a very much longer time when the soil is neutralized up to the H_2 stage than below it. As nitrites are not available to plants and may even be toxic, the sudden fall in the crop yield might be partly due to that case.

It must be pointed out, however, that these are tentative explanations and are offered merely to point out that an extensive laboratory investigation bearing on the nature of reactions involved in the triple system, lime, soil, and manure must be undertaken before a satisfactory explanation can be offered as to the differences in the behaviour of lime in the presence of various manures.

Attention must be drawn to another important point, a disregard of which might lead to some confusion. From the graph showing the effect of lime in the presence of mustard cake, it must not be inferred that mustard cake would be useless or even harmful for calcareous soils. Excess of lime due to its greater solubility can be harmful, whereas excess of chalk (calcium carbonate) on account of its low solubility will not tend to make the soil excessively alkaline. As all excess of lime must get converted into carbonate in course of time, it is believed that this tendency towards a fall in yield will disappear.

These views are also confirmed by bacteriological studies referred to above, where they have obtained characteristic differences in the behaviour of lime and calcium carbonate, which tend to show that whereas an excess of lime adversely affects nitrification and induces nitrite formation, an excess of $CaCO_3$ has no such harmful effect.

It appears from the above that it would be perhaps safer to manure with chalk than with lime, or if lime is applied, it should be done well in advance of putting in the crop. However, *if the H_1 limit is not exceeded, then lime cannot act as toxic.* It is intended to study this interesting difference in the behaviour of lime and chalk with special reference to oil cake manures in pot cultures.

ECONOMIC CONSIDERATIONS.

We have seen that lime coupled with farmyard manure gives by far the best results on these soils. This becomes all the more striking when we take into consideration the cost of manure and increased outturn.

From Fig. 10 we can roughly calculate the cost of lime and manure for obtaining a yield of say 50 maunds per acre of *Juar* fodder : In the various plots it is as follows :—

	Rs.	A.	P.
B Plot—			
Mustard cake = 25 mds. @ Rs. 4 per maund	100	0	0
Lime = 5 mds. @ Rs. 0-12-0 per maund	3	12	0
TOTAL	103	12	0
C Plot—			
Am. sulphate = 5 mds. @ Rs. 7-1-0 per maund	35	5	0
Bone meal = 3 mds. @ Rs. 5-0-0 per maund	15	0	0
Lime = 35 mds. @ Rs. 0-12-0 per maund	26	4	0
TOTAL	76	9	0
D Plot—			
Cowdung = 200 mds. @ Rs. 12-8-0 per 100 maund	25	0	0
Lime = 10 mds. @ Rs. 0-12-0 per maund	7	8	0
TOTAL	32	8	0

Another striking point about farmyard manure is that, by increasing the lime dose from 10 to 38 maunds to the acre, a 100 per cent. increase in yield is obtained ; whereas with other manures such an extraordinary increase in crop yield has not been noticed.

DETAILED DESCRIPTION OF THE METHOD OF FINDING H_1 LIMIT OF ACID SOILS.

The soil is measured in a capsule 20 c. c. capacity and shaken with about 100 c. c. of water for few minutes in a conical flask ; excess of neutral $CaSO_4$ added, also 5-10 drops of 1 per cent. sol. of Brom Thymol Blue indicator. It is then tritrated with $\frac{N}{30}$ lime solution, till a blue colour is obtained, on leaving the flask for a couple of minutes. Each c. c. of lime solution used represents 1 maund (=82 lb.) of lime $(Ca(OH)_2)$ per acre up to 6″ depth. It is not necessary to use lime water for these

titrations. In fact it is more convenient to use $\frac{N}{10}$ Baryta. It is better to start a number of soils (about a dozen or more at a time) and complete the titration in about a couple of hours or longer if time permits. The first blue colour produced soon fades to yellow on shaking and leaving and more alkali is required to produce more or less permanent bluish green. The reaction between acid soils and alkalies is very slow and a titration may even take 3 days for completion. However, it was found that, if the titration is finished at permanent blue colour which persists for about an hour, it slowly comes back to green (neutral) colour in about 3 days. That is why it is recommended that the tritration should be carried on till a blue colour is obtained that persists for some time.

SUMMARY.

1. The experimental evidence adduced so far in this investigation lends support to the view that soil acidity is due to insoluble colloidal acids or " acidoids " which are associated with surface active hydrogen and basic actions.

2. A number of reactions characteristic of soluble acids were studied in fully unsaturated soils obtained by exhaustive treatment with $\frac{N}{20}$ HCl. The outstanding facts that emerge from this study are that acidoids, though insoluble, show reactions similar to those of soluble acids. These, however, take place in three distinct stages in which one, two or three equivalents of H can take part, i.e., H_1, H_2 and H_3.

3. The experimental facts adduced so far can be best interpreted by supposing that the acidoid in a fully unsaturated soil is a tribasic acid.

4. The results of liming experiments on a two acre plot carried out at Dacca (Bengal) show that toxicity of acid soils is confined to H_1 only, for the measurement of which a simple method is outlined.

Memoirs of the Department of Agriculture in India

Studies in Soil Colloids, Part II

Factors influencing the Dispersion of Soil Colloids in Water

BY

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STUDIES IN SOIL COLLOIDS, PART II.

FACTORS INFLUENCING THE DISPERSION OF SOIL COLLOIDS IN WATER.

BY

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In an earlier publication[1] it was pointed out that the condition of the soil characterized as " good tilth " is associated with the state of aggregation of the individual particles into crumbs or compound particles. Variations in soil tilth are chiefly brought about by soil colloids particularly with reference to their flocculated or deflocculated condition. For instance, in a general way we know that hard impervious heavy soils go into suspension readily when shaken with water, whereas soils having good tilth rarely show much suspended matter when similarly treated. It is, therefore, clear that for developing a general theory of soil tilth, we must first of all establish the principles underlying the dispersion of soil colloids. This will not only enable us to correlate soil tilth with easily determinable factors, but would help in the clearer understanding of the mechanism of the newer methods of mechanical analysis that aim at attaining maximum dispersion of soil particles.

From a comparative study of the methods of preparation of the soil for mechanical analysis,[2] it was concluded that to obtain maximum dispersion of the soil, treatment with dilute HCl is necessary. This has been confirmed by Davies and Middleton.[3] These authors have enunciated the principle underlying the acid treatment as follows :—" The colloidal material is highly adsorptive and adsorbs from the soil solution many complex ions which tend to neutralize the charges on the individual particles so that they tend to coalesce and form aggregates. It is necessary to remove the adsorbed ions and substitute an ion which will impart the same charge to all particles, thus tending to make them separate, since like charges repel each other. The function of the acid treatment is to remove the adsorbed ions so that when alkali is added later, the colloidal particles may all be dispersed ".

There are two serious difficulties in the way of accepting the above statement as representing the true state of affairs :—Firstly, if the stability of the colloidal

[1] Puri and Keen. *Jour. Agri. Sci.*, 15, p. 147 (1925).
[2] Puri and Amin. *Agri. Res. Inst. Pusa Bull.* 175 (1928).
[3] Davies and Middleton. *Proc. Int. Cong. Soil Sci.*, Vol. 1 (1928).

suspension is merely brought about by the fact that all the particles have the same "adsorbed" ion, then the dispersion should be complete whatever hydroxide is used after the preliminary acid treatment. This is definitely not the case as we shall see later. Secondly, as the soil colloids are known to "adsorb" chiefly electropositive ions under ordinary circumstances, it cannot matter very much whether they are similar or dissimilar, as long as the particles remain negatively charged and there is no evidence of a reversal of charge in the case of soil colloids. In fact, as will be shown in Part III of these studies[1], the directly determining factor in the flocculation of soil colloids is not so much the nature of the "adsorbed" ion as that of the ion in solution, and therefore it is not very material what the nature of the adsorbed ion is as long as the particles remain negatively charged.

In the process of drying of soil colloids, as it happens in nature, the water film gets smaller and smaller as evaporation proceeds and the adjacent particles are drawn together on account of the surface tension of the film water and ultimately get wedged together forming compound particles which can be torn asunder by using considerable mechanical force. When these particles come in contact with water again, they have a tendency to pass into suspension; this is opposed by the cohesion of the cemented particles; and in the absence of electrolytes the ultimate state of affairs is determined by the equilibrium condition of these opposing forces : if the dispersion force is stronger than the cohesive power, the colloid will pass into suspension, otherwise not. In what follows an attempt will be made to find out the influence of various factors on the dynamics of this equilibrium.

In Part I of these studies[2] evidence was brought forth in support of the general theory of soil acidity as being due to insoluble colloidal acids ("acidoids") and the so-called adsorption of bases was ascribed to "saloid" formation. Although, through force of habit and a natural tendency of uniformity of expression, the term "adsorption" has been freely made use of even by those workers who no longer believe in the phenomenon of adsorption as being radically different from chemical reaction, precision will be gained if we restrict the use of adsorption in the case of soils to moisture intake from the vapour phase and make more frequent use of chemical terminology. Thus we can designate a completely unsaturated clay as clayic acid (Michaelis "acidoid") and its salts as clayates. As has been shown elsewhere,[3] clayic acid may be regarded as a tribasic acid which forms normal as well as acid salts. This terminology will be freely used in this paper.

EXPERIMENTAL.

Various methods of dispersion without the use of chemicals have been suggested from time to time, such as simple shaking with water for a number of hours, rubbing, churning, boiling, etc.

[1] Puri, A. N.   Flocculation of soil colloids.   *Mem. Dept. Agri. India, Chem. Ser.*, Vol. XI, No. 6.
[2] Puri, A. N.   Studies in Soil Colloids, Part I.   Base Exchange and Soil Acidity.   *Mem. Dept. Agri. India Chemical Series*, Vol. XI, No. 1.
[3] Puri, A. N.   Studies in Soil Colloids, Part I.   loc. cit.

None of these, however, is reproducible in the hands of different workers. These were, therefore, discarded in favour of simple contact with water for 24 hours. This is the nearest approach to field conditions and the results are strictly reproducible, and although in a given field the values might vary from time to time, this is exactly what may be desirable to know.

Throughout this investigation the degree of dispersion was measured by determining the percentage of conventional clay (0·002 mm. diameter) by the pipette method after leaving the soil in contact with water for 24 hours (referred to as dispersion factor or D. F.) and expressing it as percentage on the total clay content of the soil obtainable on complete dispersion. This is referred to as Dispersion Coefficient (D. C.) or D. C. $= \frac{\text{D. F.} \times 100}{\text{clay content}}$. In other words, dispersion coefficient measures the percentage of the total clay that can pass into the suspensoid state by simple contact with water ; and its value varies from zero to 100 per cent., depending as to whether the soil is completely flocculated or completely dispersed.

The exact procedure for the determination of the dispersion factor is as follows :—

10 grams of soil are made up to one litre with water and left overnight in a cylinder or bottle of uniform diameter, after mixing the soil and water by turning end over end few times, taking care that the suspension is not subjected to any violent agitation which might break up the crumbs.

Next morning the suspension is thoroughly mixed by gentle end over end motion and allowed to settle for appropriate time and clay (0·002 mm.) determined by pipetting from the proper depth exactly as in the pipette method.

The total clay obtainable on complete dispersion of the soil was determined by the (Na Cl—NaOH) method developed in this laboratory.[1]

The dispersion of clayic acid and various metallic clayates.

The formation of metallic clayates can be accomplished in two ways :—

(a) Exhaustive treatment of the natural clay with a neutral salt when the various clayates are converted into a single clayate by double decomposition.

(b) Conversion of the natural clay into clayic acid by treatment with $\frac{N}{20}$ HCl, and then the formation of various clayates by neutralizing with different hydroxides.

It must be remembered that by the first method we can only get di-hydrogen metallic clayates, whereas by the second we can have any acid or normal saloid.[2] For comparison of dispersion, therefore, di-hydrogen saloid was studied in both cases.

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[1] Puri, A. N. A new method of dispersing soils for mechanical analysis *Mem. Dept. Agr. India, Chemical Series,* Vol. X, No. 8.

[2] Puri, A. N. Studies in Soil Colloids, Part I. loc. cit.

In Table I are given values for the dispersion factor as well as the dispersion coefficient for three soils with various ions introduced by exhaustive treatment with their chlorides, the soils being subsequently washed and air dried. In every case 10 grams of air-dry soil were made up to one litre and the dispersion factor determined as described above.

TABLE I.

Dispersion of clayic acid and various clayates. (di-hydrogen saloids.)

Soil				Acidoid or saloid			Clay per cent.	D. F. per cent.	D. C. per cent.
Hoos Fallow	.	.	.	Clayic acid	.	.	33·9	1·0	2·9
18—27″	.	.	.	Na Clayate	.	.	35·56	34·9	98·1
(Rothamsted)	.	.	.	K ,,	.	.	30·8	3·1	10·1
				Ca ,,	.	.	31·1	3·6	11·6
				Ba ,,	.	.	32·1	3·7	11·5
				Al ,,	.	.	32·3	1·7	5·3
Dharwar soil	.	.	.	Clayic acid	.	.	60·5	1·4	2·3
				Mg. clayate	.	.	60·5	3·2	5·3
				Ca ,,	.	.	60·5	2·2	3·6
				Ba ,,	.	.	60·5	2·6	4·3
				Al ,,	.	.	60·5	0·5	0·8
Akola soil .	.	.	,	Clayic acid	.	.	60·6	2·5	4·1
				Ca clayate	.	.	60·6	3·0	4·9
				Ba ,,	.	.	60·6	3·2	5·3

A glance at Table I will show that, except in the case of Na clayate, the dispersion coefficient is very low; in other words, Na clayates go into suspension after drying by simply keeping them in contact with water, whereas other clayates require some mechanical force to assist in the dispersion. There is, however, no reason to believe that all saloids, in the absence of flocculating agents, will not go into suspension, provided the duration and extent of mechanical force is sufficient to overcome the force of cohesion that is binding the individual particles together.

The difference in the behaviour of various metallic clayates is also shown in a striking manner in an experiment where the saloid formation was effected by treating clayic acid with various hydroxides. 500 grams. of Akola soil (a heavy black cotton soil) were converted into the acidoid state by exhaustive treatment with

$\frac{N}{20}$ HCl. The soil after washing free from excess of acid was fractionated by sedimentation and clay portion (0·002 mm.) separated. To 750 c.c. lots of this clay suspension, which required 30 c.c. of $\frac{N}{10}$ alkali for neutralization (pH 7), were added 3 milli-equivalent of various hydroxides and volume made up to a litre. The suspension was then subjected to ultra-mechanical analysis. The results given in Table II show that Na and Li clayates contain the largest proportion of particles that show no sign of settling even after 19 days up to 5 cm. depth.

TABLE II.

Effect of various hydroxides on the dispersion of clayic acid.

Hydroxide							WEIGHT OF RESIDUE FROM 50 C. C. SUSPENSION PIPETTED WITH APPROPRIATE TIME/DEPTH RATIO.			
							0·002 mm. (diameter)	0·001 mm.	0·0005 mm.	19 days settling 5 cm. depth
Nil	.	.	.	.	.	.	·1620	·1156	·0570	·0100
LiOH	.	.	.	.	.	.	..	·1672	·1594	·1430
NH$_4$OH	.	.	.	.	.	.	·1668	·1518	·1455	·1024
NaOH	.	.	.	.	.	.	·1700	·1655	·1570	·1436
KOH	.	.	.	.	.	.	·1722	·1463	·1208	·0644
Mg(OH)$_2$	.	.	.	.	.	.	·1762	·1119	·0600	·0062
Ca(OH)$_2$	.	.	.	.	.	.	·1682	·1118	·0570	·0084
Sr(OH)$_2$	.	.	.	.	.	.	·1720	·1223	·0550	·0090
Ba(OH)$_2$	.	.	.	.	.	.	·1740	·1170	·0550	·0073

It appears from Table II that 84·4 per cent. of the material in Na clayate is colloidal as compared with 61·4 per cent. in the case of ammonium saloid.

It is to be remembered that the above noted differences are only shown when the soil colloids are air dried, and if a soil suspension at maximum dispersion is converted into any saloid or acidoid (with the possible exception of saloids of Al and Fe), then the dispersion remains at its maximum, provided the soil is not allowed to get dry. For this purpose Akola soil in the acidoid state was treated with the requisite amount of NaOH and brought into a state of maximum dispersion. It was then treated exhaustively with N solution of various chlorides, as well as $\frac{N}{20}$ HCl. After washing with water till free from Cl ions, it was shaken with water and made up to a definite volume. The results given in Table III show that, except

in the case of Al and Fe clayates which show slight reduction, the dispersion is practically the same and at its maximum.

TABLE III.

Dispersion of various clayates prepared from Na clayate and not allowed to dry.

Acidoid or saloid of										CLAY PER CENT.	
										5 per cent. suspension	1 per cent. suspension
Acidoid	.	.	.	.	.	.	.	.	.	60·3	54·6
Na	.	.	.	.	.	.	.	.	.	61·7	59·4
Mg	.	.	.	.	.	.	.	.	.	62·2	55·0
Ca .	.	.	.	.	.	.	.	.	.	62·1	58·0
Sr .	.	.	.	.	.	.	.	.	.	62·2	55·1
Ba .	.	.	.	.	.	.	.	.	.	63·8	59·8
Al .	.	.	.	.	.	.	.	.	.	54·5	52·4
Fe .	.	.	.	.	.	.	.	.	.	53·9	46·7

The effect of concentration of suspension on dispersion is also seen in Table III. This point will be examined in greater detail later on.

The fact that there is no change in dispersion, whatever the nature of the ion in clay, is of some importance ; for it shows that an alkali soil (rich in sodium clayates) can be successfully reclaimed after the gypsum treatment *only if it is allowed to dry and rewetted.* In this connection Kelly has rightly stated : " The high dispersion of leached alkali soil is not easily overcome. In addition to replacing the sodium by calcium, the element of time seems to be important. With certain alkali soils a state of pronounced deflocculation still remains after the sodium exchange components have been converted into calcium compounds........... Allowing the soil to dry out and the use of flocculents may therefore be very helpful in the reclamation of an alkali soil ".[1]

The progressive production of Na clayates from clayic acid as effecting dispersion is shown in Table IV.

The following soils were used in 1 per cent. suspension :—

    (1) A. T. 7 Clay—12 per cent.
    (2) A. T. 4 Clay—20·9   ,,
    (3) A. T. 5 Clay—22·6   ,,
    (4) A. T. 16 Clay—32·9   ,,
    (5) A. T. 17 Clay—39·8   ,,

[1] W. P. Kelley. *Proc. First Int. Cong. Soil Sci.*, Vol. 4, p. 483, Washington (1928).

TABLE IV.

Effect of the progressive production of Na clayate from clayic acid on dispersion.

Concentration of NaOH mg. per 100 c. c.							DISPERSION COEFFICIENT (PER CENT.)				
							(1)	(2)	(3)	(4)	(5)
0	.	.	.	.	.	.	10·3	7·6	4·0	8·7	8·3
1·33	.	.	.	.	.	.	13·5	8·6	3·5	13·1	..
2·66	.	.	.	.	.	.	21·8	24·9	11·1	19·7	14·9
3·99	.	.	.	.	.	.	66·6	28·7	28·8	20·7	21·4
5·32	.	.	.	.	.	.	58·3	61·2	50·9	55·4	27·4
5·98	.	.	.	.	.	.	..	65·1	..	64·4	31·5
6·65	.	.	.	.	.	.	92·0	70·8	68·6	68·6	36·0
7·31	.	.	.	.	.	.	..	..	78·7	76·6	41·4
7·98	.	.	.	.	.	.	92·0	87·1	88·5	80·6	46·3
9·31	.	.	.	.	.	.	97·5	100	93·8	91·9	55·0
10·64	.	.	.	.	.	.	99·0	..	94·7	100	65·4
11·97	.	.	.	.	.	.	100	..	98·6	..	73·4
13·33	.	.	.	.	.	.	..	..	100	..	84·9
14·66	.	.	.	.	.	.	..	..	..	..	82·3
15·99	.	.	.	.	.	.	..	..	..	..	90·9

A comparison of the progressive dispersion action of clayate formation with various hydroxides of alkali metals is shown in Table V, in the case of Akola soil (clay content 60·32 per cent.). The results plotted in Fig. 1 con-
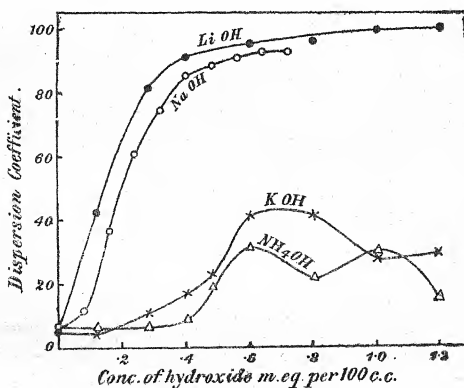
Fig. 1.

firm the previous observation that Li and Na clayates disperse much more readily than any other clayate.

TABLE V.

Effect of various hydroxides on the dispersion of clayic acid.

CONCENTRATION OF HYDROXIDES							DISPERSION COEFFICIENT			
Milli-eq. per 100 c. c.							LiOH	NaOH	NH$_4$OH	KOH
0	.	.	.	.	.	.	5·5	6·2	5·6	5·5
0·08	.	.	.	.	.	.	..	11·8	..	..
0·12	.	.	.	.	.	.	44·1	..	6·4	4·5
0·16	.	.	.	.	.	.	..	36·7	..	..
0·24	.	.	.	.	.	.	..	60·5	..	..
0·28	.	.	.	.	.	.	83·7	..	..	..
0·32	.	.	.	.	.	.	..	74·6	6·7	10·8
0·40	.	.	.	.	.	.	92·4	86·6	..	..
0·48	.	.	.	.	.	.	92·9	89·5	8·5	17·2
0·56	.	.	.	.	.	.	..	91·4	9·7	23·5
0·60	.	.	.	.	.	.	95·0	..	..	..
0·64	.	.	.	.	.	.	..	93·0	31·8	41·3
0·72	.	.	.	.	.	.	..	93·0	..	..
0·80	.	.	.	.	.	.	96·3	..	21·4	41·2
1·00	.	.	.	.	.	.	99·8	..	29·4	28·4
1·20	.	.	.	.	.	.	100	..	15·3	34·6

The fact that only the Li and Na clayates give a high dispersion coefficient is very important from the practical point of view, as it affords a very simple method of determining how far the soil colloids exist as Na clayates in nature. The value of this will be readily recognized in the light of recent researches on alkali soils notably those of Sigmond, Gedroiz, Joffe, McLean, Kelley and Scofield ; which leave no doubt as to the fact that alkali soils are rich in exchangeable Na, or in other words, contain a high percentage of Na clayates.

A number of natural soils were examined ; some of these were soils rich in exchangeable Na. The dispersion coefficient as well as permeability to water (determined by the writer's cylinder method[1]) for these soils is recorded in Table VI.

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[1] Puri. A. N. A new percolating cylinder and some of its uses. *Agri. Jour. India*, XXIV, Pt. 6.

TABLE VI.

Dispersion of various soils.

Soil	Clay per cent.	D. F. per cent.	D. C. per cent.	Percolation c. c. per hour
Highly acid soils—				
(1) Thuravoor. East kan (Madras) .	59·8	1·9	3·2	142
(2) Dacca (Bengal)	35·9	3·4	9·5	172
(3) Punja lands Kuppapuran (Madras) .	25·4	1·0	3·9	180
Agricultural soils of varying fertility—				
(*a*) Unirrigated—				
(1) Akola (Bombay)	51·6	1·5	2·9	16
(2) Malabar Coast (Madras) . .	21·8	1·3	5·9	22·5
(*b*) Irrigated soils—				
(3) Karnal (Punjab)	29·6	4·4	14·9	23
(4) Naryan I	9·2	1·3	14·1	15
(5) „ II	9·1	1·1	12·1	16
Calcareous soils—				
(1) P. 11 (Pusa)	17·8	5·3	29·8	13
(2) P. 3 (Pusa)	9·4	2·7	28·7	16
(3) Soil from Turky (Bihar) Infertile .	19·1	11·8	61·7	..
Alkali soils—				
(1) Sir Ganga Farm (Poor crop) . .	9·0	3·4	38·2	7
(2) Bara (Untreated) Infertile 1st. ft. .	20·3	15 8	77·8	1
(3) „ „ „ 2nd ft. .	14·0	9·3	66·6	1
(4) Montgomery (infertile) . . .	74·3	75·7	100	0
(5) Typical Bara („) . . .	21·2	19·4	91·3	1
(6) Bara Gypsum treated still infertile 1st ft.	15·1	11·6	76·6	1
(7) Bara Gypsum treated still infertile 2nd ft.	14·3	7·2	50·1	1
(8) Bara Gypsum treated crops doing well 1st ft.	16·0	2·9	18·1	42
(9) Bara Gypsum treated d ing well 2nd ft.	14·8	3·6	24·1	46
(10) Fair land in Bara tract . . .	4·4	1·2	27·3	27

A glance at Table VI will reveal some interesting facts :—

(1) Alkali soils rich in exchangeable Na show a high dispersion coefficient and a very low rate of percolation.

(2) Successful treatment with gypsum increases the permeability and is reflected in a lowering of the dispersion coefficient.

(3) Highly acid soils give a very low dispersion coefficient and a very high rate of percolation.

(4) Irrigated soils give a comparatively higher dispersion coefficient than unirrigated ones.

A method of preliminary treatment of the soil for mechanical analysis based on the easy dispersibility of Na clayates has been developed.[1] Briefly, it consists in treating the soil with N. NaCl when the clay is converted into the Na saloid and the soil disperses completely. The method is applicable to calcareous soils which shows that an excess of $CaCO_3$ does not prevent the soil from accumulating exchangable Na. This is shown in a striking manner in the case of an infertile Bihar soil (Table VI) which contains 16 per cent. $CaCO_3$. Here also the infertility is associated with a high dispersion coefficient. The fact noted above leads to a very important conclusion, i.e., an alkali soil (rich in exchangeable Na) cannot be permanently improved by adding lime to it. This latter point will be discussed in part III of these studies.[2]

Determination of the dispersion coefficient affords a simple means of finding the degree of alkalization of a given soil. It gives at once the state of aggregation of the particles and consequently some idea of the soil tilth. Periodic determinations of this value on lands recently brought under canal or well irrigation will show whether there is likelihood of soil deterioration.

Although dispersion coefficient cannot be regarded as a definite constant in the sense that it could be used for soil characterization like, for instance, the clay content, its variations in a given soil are not very great ; in fact, it is these variations a knowledge of which would be helpful in solving many difficult problems connected with soil tilth.

We have seen that all good soils give a comparatively low dispersion coefficient, whereas all barren alkali soils show a high value ; and what is more important, where the soil has been improved by gypsum treatment, the dispersion coefficient has gone down and where it has not responded to this treatment the value is still comparatively high. Colloids are considered reversible whose solutions leave a residue soluble in water when desiccated at ordinary temperature ; and irreversible, which under the same circumstances yield a residue insoluble in water. There is no doubt that all soil colloids would be classed as reversible, but the manner in which they can be brought back into solution is not the same. For instance,

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[1] Puri. A new method of dispersing soils for mechanical analysis. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. X, No. 8.

[2] Puri. Studies in soil colloids, Part III. Flocculation of soil colloids. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. XI, No. 6.

calcium saloid if once dried will not go into suspension unless assisted by mechanical force to overcome cohesion, whereas in the case of sodium and lithium saloids, no mechanical force is required ; and we are dealing with phenomenon treated by Donnon[1] who has shown that under certain assumptions the mutual reactions of the forces of cohesion and the forces of molecular attraction can result in an " auto-subdivision " of the substance which ceases before molecular dimensions are reached.

Mattson[2] who studied the dispersibility of soil saturated with various bases also arrived at the conclusion that sodium hydroxide causes a higher degree of dispersion than either potassium or ammonium hydroxide. Similar conclusions have been arrived at by Thomas.[3]

The complete reversibility of sodium clayates after drying and their remarkable property of " auto-subdivision " on coming in contact with water is made the bases of a simple test for alkali soils rich in exchangeable sodium. The test is carried out by working the soil into a pallet with a little water and air drying. The dried pallet when placed in a shallow basin (e.g., a petri dish) of water, develops a turbid ring round it within a minute or two which covers the whole surface of water in ten to fifteen minutes. The test is extremely simple and can be carried out actually on the field.

Effect of concentration of suspension on dispersion.

The mutual influence of particles on one another at the time of settling is an important consideration in all studies of dispersion. It is therefore of interest to know the minimum concentration beyond which this influence begins to be felt, as well as the probable nature of this effect.

According to Odén it is not safe to go much beyond 1 per cent., whereas Wiegner maintains that a concentration of more than 5 per cent. can be used without introducing any error.

It was decided to study this effect in the case of completely dispersed suspensions containing one kind of exchangeable ion in the clay complex. Some of these results have been recorded in Table III, which shows that in all cases a higher clay content is obtained at 5 per cent. suspension of the soil than at 1 per cent., but this difference is less in the case of Na clayate than in others. In order to study the point in greater detail, a number of concentrations were examined in the case of clayic acid and Na clayate suspensions. The results given in Table VII throw light on the nature and extent of this influence. The effect is obviously due to the increase of viscosity brought about by an increased concentration of soil colloids, so that the rate of settling is decreased thereby. However, it is to be noted that the effect is not very great even in 5 per cent. suspensions of soil (equivalent to 3 per cent. suspension of clay), especially in the case of Na clayate soil.

[1] *Zeitschr, F.   Phys. Chem.*, 1901, Vol. XXXVII, p. 735, also 1903, Vol. XLVI, p. 197.
[2] Mattson, S.   *Proc. First Int. Cong. Soil Sci. Commission*, II, p. 105, Washington, 1928.
[3] Thomas, M. D.   *Soil Sci.*, 25, p. 419 (1928).

TABLE VII.

Effect of concentration of suspension on dispersion of clayic acid and Na clayate.

Concentration of soil suspension	PERCENTAGE OF PARTICLES				
	Na clayate	Clayic acid			
	·002 mm. per cent.	·002 mm. per cent.	·001 mm. per cent.	·0005 mm. per cent.	10 days settling 5 cm. depth
0·25	..	57·6	54·4	52·2	40·8
0·5	..	56·8	57·0	51·6	44·4
1·0	59·8	55·1	54·6	51·7	45·1
2·0	59·9	..	..	..	..
2·5	..	55·7	53·4	51·3	46·2
4·0	60·7	..	..	..	..
5·0	..	58·2	55·3	52·1	47·7
6·0	62·5	..	..	..	..
8·0	64·7	..	..	..	..
10·0	67·9	69·7	64·1	58·4	55·1
13·33	76·6	..	..	..	..

Recently it has been shown by Shaw and Winterer[1] that the sedimentation methods of mechanical analysis are subject to a fundamental error, which results in an uneven distribution of settling particles in the sedimentation column. They ascribe this phenomenon to the neutralization of the negative charge of the particles by the positive charge of the walls of the container, causing them to coalesce with one another and settle down faster. Whatever reluctance one might feel in accepting the explanation of the phenomenon offered by the authors, the fact remains, which, as they themselves point out, is either due to a defective technique or to an inherent defect in the method; if the latter, then all methods of dispersoid analysis must be influenced in a similar manner. It became necessary therefore to study this point using the pipette technique employed throughout this investigation.

2·5 per cent. suspensions of Akola clayic acid and Na clayate soil were used, both being at their maximum dispersion. They were contained in glass cylinders of 6″ diameter. Particles of ·002 mm. limiting diameter were pipetted off with a 50 c. c. pipette having its tip bent at right angle, and evaporated to dryness. After

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[1] Shaw and Winterer. A fundamental error in mechanical analysis of soil by the sedimentation method. *Proc. Int. Cong. Soil S.i.*, Vol. I, p. 385 (1928).

each sampling the suspension was shaken again and the process repeated. Altogether 10 samples were taken : 5 by keeping the tip of the pipette in the centre and 5 near the wall of the container.

The results given in Table VIII leave no doubt that, whatever the distribution of the particles might be after settling at the bottom, the process of settling is uniform and vertical and the concentration of the particles is the same at every point in a transverse section of a settling column.

TABLE VIII.

Transverse distribution of particles in a settling column.

Sample No.								Weight of residue in each sample of 50 c.c.			
								Na clayate soil		Clayic acid soil	
								Side	Centre	Side	Centre
1	.	.	.	.	.	.	.	·7430	·7420	·7090	·7010
2	.	.	.	.	.	.	.	·7474	·7420	·7030	·6965
3	.	.	.	.	.	.	.	·7580	·7633	·7195	·7274
4	.	.	.	.	.	.	.	·7567	·7630	·7280	·7296
5	.	.	.	.	.	.	.	·7535	·7600	·7387	·7382
Mean	.	.	.	.	.	.	.	*·7517*	*·7538*	*·7196*	*·7185*

SUMMARY.

(1) Factors influencing the dispersion of soil colloids in water have been studied with particular reference to the nature of the exchangeable ion in them.

(2) When a soil is left in contact with water for 24 hours and clay (·002 mm.) determined by the pipette method, the nearest approach to the state of aggregation as it exists in the field is obtained and the value thus obtained is designated as dispersion factor (D. F.).

(3) Dispersion coefficient (D. C.) is defined as the percentage of the total clay that can pass into suspension by "auto-subdivision" on being left in contact with water for 24 hours.

i.e., D. C. $= \dfrac{\text{D. F.} \times 100}{\text{Clay content}}$

(4) Dispersion coefficient can be used for comparing the state of aggregation of particles in different soils and in general shows a positive correlation with the degree of alkalization in a given soil. All barren alkali soils, rich in exchangeable Na, studied, had a dispersion coefficient above 50 per cent. Successful treatment of an alkali soil with gypsum results in a substantial lowering of the dispersion coefficient and where the response is poor, the change in this value is slight.

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Memoirs of the Department of Agriculture in India

Feeding Experiments at Hosur, 1926, 1927 and 1928

BY

F. J. WARTH, M.Sc.,

Physiological Chemist, Imperial Department of Agriculture

IMPERIAL INSTITUTE OF AGRICULTURAL RESEARCH, PUSA

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FEEDING EXPERIMENTS AT HOSUR, 1926, 1927 AND 1928.

BY

F. J. WARTH, M. Sc.,

Physiological Chemist, Imperial Department of Agriculture.

I. INTRODUCTION.

The work reported in this paper is the result of a co-operative effort in which the joint resources of the Nutrition Section and the Madras Department of Agriculture were utilized to carry out animal nutrition experiments. The proposal originated from the Nutrition Section, it was accepted by the Director of Agriculture, Madras, and the Deputy Director of Agriculture, Livestock. Madras, was given a free hand to co-operate with the Nutrition Section. The Madras Department provided the animals and all the food-stuffs used in the tests, the accommodation for the experiments and many other facilities. The work was organized and carried out by the Nutrition Section. I wish to record here my sincere thanks to Mr. R. W. Littlewood, Deputy Director of Agriculture, Livestock, for the whole hearted help he has given to the Nutrition Section at all times throughout the course of the experiments.

I have also to acknowledge the help given by my staff. Various members assisted in the supervision and management of the digestion and feeding tests, as well as in carrying out all the analytical work.

Object of experiments.

The object of the experiments was to study the nutritive value of spear grass fodders. This grass is very common on many pasture areas in India. It is also converted into fodder and hay in large amounts and is baled and transported from place to place. Owing to its wide distribution and extensive use, it certainly deserves study. The Hosur Cattle Breeding Farm produces a representative natural growth of spear grass herbage and is therefore eminently suitable for the enquiry it was proposed to carry out.

The following products have to be considered :—

  (*a*) *The standard product Prime Spear grass hay.*—This suffers from the grave disadvantage that it is full of spears (the awns of the seed).

[53]

(b) *Mature hay, 80 per cent. spears removed.*—The Madras Department showed that the spears could be removed effectively and economically by rake if the grass were allowed to mature. Hence the second common and now widely used fodder—mature hay, spears removed.

(c) *Early cut hay.* The Madras Department made repeated efforts to save early cut hay. In suitable seasons excellent early cuts have been obtained at Hosur.

(d) *Spear grass silage.* Silage making from spear grass at different stages of development has been carried out by the Madras Department at Hosur.

The Nutrition Section carried out tests with each of these products.

General procedure.

Two distinct lines of work were undertaken. In one series of tests very accurate digestion experiments were made with mature animals. The question here was simply to obtain reliable information regarding the digestibility of the fodders. The other series consisted of feeding experiments over extended periods to determine the live weight increase and nutritive effect of spear grass fodders.

II. DIGESTION EXPERIMENTS.

Procedure.

Three products were tested, namely, spear grass silage, mature spear grass hay (80 per cent. spears removed) and early cut spear grass hay. Six bulls were selected for this work and each bull was fed with the three fodders in succession. Hence six separate and parallel sets of digestion data were obtained for each fodder. Both concentrate and coarse fodder were fed in proportion to live weight, the coarse fodder being provided in slight excess to ensure consumption of the desired amount. In the case of silage and young hay, the desired consumption was satisfactorily attained. In the case of mature hay every animal ate less than its allotted amount. The consumption was, however, quite regular. For each ration a preliminary period of feeding preceded the actual digestion experiment which lasted twelve days.

Digestibility of the mixed ration.

The results of these trials are shown below :—

TABLE I.

Digestion co-efficients of the mixed ration.

	Food consumed—in grams dry matter per day			Live weight lb.	Consumption per 1,000 lb. live weight	Nutritive ratio	Percentage protein in ration	Ratio R/C	Digestion Coefficients				
	Rough-age	Concen-trate	Total						Dry matter	Organic matter	Protein	Ether extract	Carbo-hydrates
Silage	6,087	544	6,631	928	7,146	12·04	10·55	11·19	59·7	61·0	53·1	60·1	66·4
	5,406	460	5,866	736	7,970	10·23	10·37	11·76	58·0	62·5	45·5	54·0	64·7
	5,498	441	5,939	719	8,072	10·82	10·18	12·47	57·2	61·6	45·3	56·3	62·2
	5,847	497	6,344	837	7,579	10·18	10·38	11·76	60·0	64·4	49·3	60·9	66·4
	5,183	431	5,614	769	7,300	9·00	10·30	12·10	56·8	61·4	47·0	57·6	63·1
	4,693	394	5,087	648	7,850	10·82	10·33	11·92	57·3	61·8	45·4	54·3	63·8
Average	5,453	461	5,914	..	7,683	10·55	10·40	11·53	58·2	62·7	47·8	57·2	64·4
Mature hay.	5,142	537	5,679	928	6,110	11·19	7·49	9·58	53·9	57·3	59·0	61·5	57·1
	4,575	453	5,028	736	6,838	12·36	7·27	10·08	52·3	50·9	53·9	61·0	56·1
	4,421	435	4,856	719	6,752	12·19	7·25	10·17	53·0	56·9	53·7	58·8	57·0
	4,843	490	5,333	837	6,395	11·72	7·35	9·92	53·0	56·8	56·6	57·8	56·3
	4,458	426	4,914	769	6,390	12·22	7·10	10·55	51·9	55·6	54·1	59·1	55·5
	3,603	389	4,052	648	6,251	11·90	7·56	9·43	51·5	55·0	55·9	61·6	55·0
Average	4,525	455	4,980	..	6,457	11·84	7·34	9·96	52·0	56·2	55·5	58·8	56·2
Young hay.	5,503	528	6,031	928	6,499	7·69	10·75	10·43	58·7	63·0	61·0	67·0	62·8
	5,303	446	5,749	736	7,811	7·98	10·32	11·89	57·0	61·8	60·9	61·8	61·7
	5,314	428	5,742	719	7,986	8·31	10·18	12·42	56·4	60·1	58·8	60·0	60·2
	5,534	482	6,016	837	7,191	7·97	10·43	11·47	55·5	62·0	62·2	62·6	62·0
	5,300	410	5,719	769	7,436	8·27	10·13	12·66	57·1	60·9	57·8	64·9	60·8
	4,208	382	4,590	648	7,083	7·95	10·56	11·01	58·5	62·0	61·1	64·4	62·0
Average	5,194	447	5,641	..	7,384	8·03	10·39	11·65	57·8	61·6	60·3	63·4	61·6

The figures are given in considerable detail to bring out certain remarkable regularities which have been found. The chief points to note in these results are :—

(a) *Consumption of roughage.* It is significant that silage and young hay are consumed more readily than mature hay even when limited amounts are fed.

(b) *Digestion of protein.* Protein was best digested in the young hay ration. The mature hay comes next and silage is last. It may be noted that silage has this low position in spite of the fact that the ration contained as much protein as the young hay ration and considerably more than the mature hay ration. The figures indicate that protein in silage rations is less digested than in hay rations.

(c) *Digestion of carbohydrate.* In carbohydrate digestion the silage ration is easily the best. The young hay comes next. Mature hay is last.

With regard to variations in the digestion results, a first glance shows that the digestion coefficients of any one fodder as determined from tests with six different animals agree moderately well. A careful examination shows, however, that the observed variations are not mere chance fluctuations about a mean figure. The values found are related to the ration consumed. Some animals ate relatively more roughage, others relatively less. Accordingly, the ratio of roughage to concentrate, the percentage of protein and the nutritive ratio were distinct for each animal in each experiment. It can be shown that these factors exerted a definite effect upon digestion. To bring out this point, the data have been arranged in order and instead of the result itself the numerical order of the result is recorded in Table II.

TABLE II.

Relation between digestion and the amount of roughage consumed.

No. of bull	ORDER OF R/C			Total	ORDER OF CARBOHYDRATE DIGESTION			Total
	1st test	2nd test	3rd test		1st test	2nd test	3rd test	
1	1	2	1	4	1	1	1	3
2	2	4	4	10	3	4	4	11
3	6	5	5	16	6	2	6	14
4	2	3	3	8	1	3	2	6
5	5	6	6	17	5	5	5	15
6	4	1	2	7	4	6	2	12

Relation between digestion and the amount of roughage consumed—contd.

No. of bull	ORDER OF PROTEIN IN RATION			Total	ORDER OF PROTEIN DIGESTION			Total
	1st test	2nd test	3rd test		1st test	2nd test	3rd test	
1	1	2	1	4	1	1	2	4
2	3	4	4	11	2	6	4	12
3	6	5	5	16	6	6	5	17
4	2	3	3	8	2	2	1	5
5	5	6	6	17	4	4	6	14
6	4	1	2	7	5	3	3	11

This Table contains a fund of information which cannot be fully discussed here. The following points bear directly on the subject in hand.

Bullock No. 1 consistently ate a low proportion of roughage. He scores lowest marks in ratio of roughage to concentrate. As a consequence, his ration was consistently richest in protein. It is significant that this animal has attained the best digestion of both protein and carbohydrate. The opposite case is that of bullocks 3 and 5. They consumed the highest proportion of roughage in all the tests. They have high marks in ratio of roughage to concentrate. Their rations were the lowest in protein and their digestion results are the lowest. No. 6 evidently did not come up to its average in the second test. It is interesting to note that it was somewhat off its feed at this time and its digestion results are lower than usual. The main fluctuations in the digestion coefficients are thus proved to be related to the proportion of roughage and concentrate in the food eaten. Hence the average digestion coefficients represents the digestion of the average ration with a considerable degree of accuracy. These are in fact the most consistent digestion data the writer has met.

Digestibility of the coarse fodders.

From the average results of Table I, the digestibility of the coarse fodders has been calculated by making appropriate allowance for the digested concentrate. For each fodder three sets of results are given in Table III.

TABLE III.

Composition and digestibility of spear grass fodders.

		Dry matter	Organic matter	Crude protein	Ether extract	Carbo-hydrates
Silage	Composition . .	100	87·78	6·62	1·26	79·9
	Digestion coefficients	56·96	61·67	18·62	39·73	65·21
	Amount digested from 100 lb. dry matter.	56·96	54·21	1·23	0·50	52·10
Mature hay	Composition . .	100	91·98	2·86	0·74	88·38
	Digestion coefficients	50·72	54·52	..	21·80	56·79
	Amount digested from 100 lb. dry matter.	50·72	50·14	0·19	0·16	50·19
Young hay	Composition . .	100	90·78	6·97	1·35	82·46
	Digestion coefficients	56·66	60·63	42·15	44·21	63·34
	Amount digested from 100 lb. dry matter.	56·7	55·1	2·94	0·60	51·4

The first line shows the percentage composition of the foodstuff, the second line shows the percentage digestion (digestion coefficient) of each constituent and the third line shows the amount of each constituent digested from 100 lb. of dry substance. With regard to chemical composition, the protein content is the most significant item. The young hay and silage are similar, the mature hay is much poorer. In fibre content young hay is lowest and mature hay highest. Judged by chemical composition, the young hay, which is about the best that can be produced on Hosur land, is fair in quality, and the mature hay is poor. The digestion coefficients of the three fodders show striking differences. The protein of young hay is digestible to the extent of 42 per cent. This is not a good value. The silage protein is only digested to the extent of 18·6 per cent. This is poor. It appears that conversion of a fodder into silage reduces the digestibility of protein. Similar results have been obtained in other experiments carried out by the Nutrition Section. Mature hay provides no digestible protein. In carbohydrate digestion silage is superior to the other fodders. Young hay comes second and mature hay is last.

Starch equivalents of Hosur fodders.

The writer has recently pointed out certain difficulties experienced in assigning correct starch equivalent values to Indian fodders.[1] While such doubt exists, three methods are being employed to arrive at tentative values and the results of all three methods will be recorded until further trials make it possible to select the most reliable figure. Table IV gives a summary of values found for the three typical Hosur spear grass products.

TABLE IV.

Starch equivalent values of spear grass fodders.

Fodder	100-LB. DRY SUBSTANCE		STARCH EQUIVALENT		
	Digestible crude protein	Total digestible nutrients	K. 1	K. 2	A.
Silage	1·23	54·4	32·9	33·5	40·2
Early cut hay	2·94	55·7	35·3	35·2	42·2
Mature hay	..	50·5	28·6	28·2	34·0

K. 1.— Kellner's starch equivalent by Fibre method.
K. 2.— Kellner's starch equivalent by Bangalore Graphic Method.
A.— American starch equivalent by Bangalore Graphic Method.

These digestion experiments have established two important facts :—

(*a*) The superiority of the two improved fodders silage and young hay has been established and rough quantitative measures for the superiority have been procured.

(*b*) A very important distinction between the two improved fodders has been brought to light. The silage is found to be a good succulent fodder which provides easily digestible carbohydrates. It is suitable for cows receiving ample concentrate. The young hay provides moderately easily digestible carbohydrates together with a very appreciable amount of protein. The use of this fodder for growing stock would greatly economise concentrate.

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[1] The *Journal of the Central Bureau for Animal Husbandry and Dairying in India*, Vol. III, Part IV. page 119.

III. FEEDING EXPERIMENTS.

The following statements give in summarized form the object of each trial and the results obtained.

1. Comparison of spear grass silage and prime hay for calf feeding (1926).

Two groups of eight calves were selected. All received the same concentrate allowance, *viz.*, 3 lb. a day per head. One group was fed on spear grass silage, the other on spear grass hay. The fodder was provided *ad lib.*, and each animal's daily consumption was determined. The test lasted twelve weeks. The following average results were obtained.

TABLE V.

Comparative feeding test with spear grass silage and hay.

	Silage group	Hay group
	lb.	lb.
Average roughage consumption	8·25	5·85
Initial average live weight	486	473
Final average live weight	512	472
Live weight increase	26	—1

It appears from this experiment that spear grass silage is eaten with relish. Consumption is good and leads to fair live weight increase. The spear grass hay is consumed in amounts which barely suffice for maintenance. This is the first feeding experiment with spear grass silage reported on in India. The result is satisfactory and of considerable practical significance. It should be noted that in the above experiment there were very considerable fluctuations in the consumption of silage. The accompanying chart illustrates the weekly fluctuations.

TABLE VI.

Fluctuations in silage consumption.

WEEKS.

It will be noticed that the weekly consumption tends to rise to a maximum of 30 and then fall to 24 kilo gram dry matter. Similar fluctuations were obtained in some experiments at Karnal [1] and there it was shown that fluctuations in roughage consumption were associated with a wide nutritive ratio. The nutritive ratio in the Hosur silage experiment can be calculated from digestion data which will be given later. These data show that the nutritive ratio during the peaks of high silage consumption was 9·2, whilst during the periods when consumption fell, the nutritive ratio narrowed down to 7·9. It seems probable therefore that fluctuations of consumption in this experiment are also attributable to variations in the nutritive ratio.

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[1] Feeding Experiments at Karnal. *Mem. Dept. Agri. India, Chem. Ser.* Vol. X, No. 1.

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[1] Feeding Experiments at Karnal. *Mem. Dept. Agri. India, Chem. Ser.* Vol. X, No. 1.

2. Spear grass silage as a sole ration for growing cattle (1927).

This experiment was inspired by the work at Cambridge which has shown that young grass has the nutritive value of concentrate. For the experiment at Hosur a special silage was prepared from young spear grass. The product was excellent in quality. It was relished and consumed heartily by the animals under test. As the trial progressed, it became evident that there was something wrong with the ration. The animals did not thrive. They failed to increase in live weight and their coats developed a marked roughness. After ten weeks' trial the test was abandoned. The data obtained in the test are as follows :—

TABLE VII.

Feeding test with spear grass silage as the sole food.

									lb.
Average initial live weight	.	.	.	.	.	.	.	.	444
Average final live weight	.	.	.	.	.	.	.	.	444
Average consumption of silage (Dry matter)	.	.	.	.	.	.	8·12

It is very important to realize why this ration was a failure. The daily food consumption was not particularly good, but such consumption of a well balanced ration would have yielded better results. The ration is not well balanced. Digestion experiments, which will be described later, show that the silage ration alone did not provide sufficient digestible protein. Now if such a good silage cannot provide sufficient protein, it is clear that an ordinary over-ripe hay cannot be used without concentrate. This negative result may be considered highly instructive for cattlemen in India.

3. Influence of minerals on the nutritive value of spear grass silage (1927).

The experiment described above was carried out in duplicate with two groups of eleven calves. The group already discussed did not receive minerals. The other group received a mineral supplement. During a test extending over ten weeks, this supplement showed no beneficial effect. The animals appeared to have increased 3 lb. in live weight, but this slight change in weight is not significant. They become rough coated and unthrifty in appearance exactly like the other group. In short, a mineral supplement was unable to overcome a decided protein deficiency.

4. Production value of spear grass hay and concentrate (1927).

The entire failure of the hay ration in 1926 made it desirable to repeat the trial under new conditions. In the first place, the hay was improved by removing 80 per cent. of the spears with a rake while the grass was maturing and it was fed

chaffed. In the second place, the concentrate was improved in quality. The results obtained during a feeding test of ten weeks were as follows :—

TABLE VIII.

Feeding test with mature spear grass hay and concentrate.

	lb.
Average initial live weight	439·0
Average final live weight	471·5
Increase in 70 days	32·5
Average consumption of hay (dry matter)	5·97
Average consumption of concentrate (dry matter)	2·73

The result is a great improvement on the previous year. It has to be noted, however, that hay consumption per 1,000 lb. live weight was not materially better during this second trial (the figures are 12·4 in 1926 and 13·1 in 1927). It must be admitted that digestion experiments (to be described later) do not shed any light on this improvement. The better quality of concentrate can hardly account for the difference. We are left with two factors.

(a) removal of spears.

(b) chaffing.

In what way and to what extent these two factors might affect the feeding result is not known at present. Very valuable information may be expected from a renewed study of these points.

5. Influence of minerals on the nutritive value of spear grass hay and concentrate (1927).

Experiment 4 was carried out in duplicate. Two lots of well balanced calves were used in the test. They were treated identically in every respect except that one lot received minerals and the other lot did not. The group receiving a mineral supplement increased 38 lb. per head, whilst the check group (considered above) increased 32·5 lb. No emphasis is laid on this difference at present.

6. Comparison of typical spear grass fodders (1928).

In this experiment early cut hay, mature hay and silage were compared with one another. Each fodder was tested with high and low rates of concentrate rationing and finally to obtain a check against spear grass products identical tests were made with *ragi* straw. There were thus four fodders and each was tested with two levels of concentrates allowance. Table IX gives a summary of the results obtained.

TABLE IX.

Results of feeding tests, 1928.

Foodstuffs	FOOD CONSUMPTION IN LB. DRY MATTER PER DAY			LIVE WEIGHTS IN LB.		
	Roughage	Concentrate	Total	Average live weight	Actual increase	Increase per 100 days
Low concentrate and silage .	9·15	2·51	11·66	592	51·5	61·3
Low concentrate and young hay.	9·65	2·51	12·16	588	45·0	53·6
Low concentrate and mature hay.	8·99	2·51	11·50	577	17·5	20·8
Low concentrate and *ragi* straw.	7·66	2·49	10·15	566	11·5	13·7
High concentrate and silage	9·46	3·45	12·91	626	67·0	79·8
High concentrate and young hay.	9·24	3·41	12·65	609	66·5	79·2
High concentrate and mature hay.	8·01	3·34	11·35	573	26·5	31·5
High concentrate and *ragi* straw.	7·48	3·36	10·84	594	39·0	46·4

The following points deserve notice in this Table :—

> (*a*) *Spear grass silage.* The high nutritive value for spear grass silage in 1926 is amply confirmed in the two experiments of 1928. The growth obtained is again far greater than with mature hay.

> (*b*) *Early cut hay.* The digestion experiments discussed in the first part of this paper indicated the superiority of early cut hay over mature hay. The results of the 1928 feeding experiments confirm the findings of the earlier digestion trials. The early cut hay has given much greater growth than mature hay in duplicate trials. It is an accepted fact that early cutting produces better material, but the extent of the benefit to be derived from early cutting is not known. The real value in this experiment lies in the fact that it shows the extent of the benefit. There is no doubt that the benefit is very great. Whilst mature hay yielded increases of 17·5 and 26·5 lb. in two different

experiments, the corresponding results with early cut hay were 45·0 and 66·5 lb. respectively. These experiments with early cut hay deserve serious attention. They indicate a line along which the nutrition of cattle in India can be improved.

(c) *Comparison of silage and early cut hay.* These two fodders gave identical results in one test, whilst in the other test silage was 15 per cent. better than young hay. This is a serious divergence which must be attributed to variations in the individual animals used in the experiment. Without further tests, the relative merits of these two fodders cannot be gauged and for the present we must be content to say that silage and early cut hay are both good and produce similar growth.

(d) *Comparison of mature hay and ragi straw.* This important experiment in which it was intended to compare spear grass products with a standard fodder has unfortunately failed to a large extent. There is reason to believe that *ragi* straw would yield better results under more normal conditions. In the Hosur experiments the following abnormal circumstances have to be recorded.

In the first place the *ragi* straw was fresh. According to local belief, fresh *ragi* straw is said to have deleterious effects. Whether there is any foundation in this local belief, it is impossible to say at present, but it is a fact that we had a considerable amount of scouring amongst the *ragi* straw animals in the early part of the test. In the second place, *ragi* straw was an entirely new fodder to these animals. A lengthy preliminary feeding period was, therefore, allowed for training the animals to eat this new food. It appears now that this preliminary feeding should have been still longer. This conclusion is based on numerical results which are shown in Table X.

TABLE X.

Showing daily average roughage consumption during first six weeks and following seven weeks.

		Silage	Young hay	Mature hay	*Ragi* straw
Low concentrate groups	1st period	4·29	4·34	4·14	3·29
	2nd ,,	4·04	4·40	4·01	3·64
High concentrate groups	1st period	4·54	4·27	3·67	3·10
	2nd ,,	4·09	4·26	3·63	3·67

These figures prove conclusively that *ragi* straw consumption improved materially during the latter part of the test, and there is no doubt that fodder would have produced better results if the animals had been given more time to adapt themselves to it. The consumption of young hay and mature hay was very constant. The silage consumption, however, shows a peculiarity which may be significant. In both tests there is a drop in consumption during the latter part of the experiment. Does this mean that young animals are likely to tire of heavy silage rations ? The point deserves notice. It is evident too that, if consumption had not fallen off, the silage would have produced still better growth.

IV. Digestion experiments with feeding test rations.

Digestibility trials were carried out with all the rations used in the above feeding experiments. Four to six calves were used for most of these tests and generally very concordant results were obtained. It is unnecessary to go into these figures in detail. The main facts established will be made clear from the following summary.

Table XI.

Digestion coefficients of the feeding test rations.

	Organic matter	Protein	Fibre	Total carbo-hydrate
1926. Hay and concentrate . . .	49·5	59·4	55·6	47·0
1927. Hay and concentrate . . .	52·3	63·7	52·4	49·7
1928. Young hay and concentrate . .	57·3	60·6	64·5	55·9
1928. Mature hay and concentrate . .	51·1	61·9	55·3	52·1
Average .	52·6	61·4	56·9	50·6
1926. Silage and concentrate . . .	54·1	50·4	61·2	53·3
1927. Silage alone	55·5	34·3*	69·2	58·1
1928. Silage and concentrate . . .	61·5	51·3	70·1	62·0
Average .	57·0	50·8	66·8	57·8
1928. *Ragi* straw and concentrate . .	60·9	59·6	67·7	59·7

* This figure is excluded from the average, because concentrate was not fed in this test.

The points to be noted are :—

(a) *Difference between hay and silage.* The hay and silage experiments have been grouped separately in order to show the striking differences between these two types of fodder. It will be noticed that in all parallel tests silage gives higher values for organic matter, fibre and total carbohydrates and lower values for protein digestion than the corresponding hay ration. Experiments carried out in three consecutive years with materials of varying quality have given the same indications every time. It can be safely accepted, therefore, that these are characteristic differences between silage and hay with respect to their digestibility. The relatively high digestibility of the carbohydrates of silage has been noted in other experiments [1] carried out by the Nutrition Section. This subject is now being studied more fully at Bangalore.

(b) *Difference between young and old hay.* The figures relating to young hay and mature hay are also noteworthy. Young hay is clearly differentiated by its high digestion coefficients for fibre, total carbohydrates and organic matter.

(c) *Ragi straw.* The digestion results obtained with the *ragi* straw ration must be considered remarkable. In protein digestion it is almost equal to the young hay ration of the same year, whilst in carbohydrate digestion it approaches the silage ration of the same year. It is possible that these excellent digestion results are partly attributable to low consumption which favours digestion, but the high values cannot be explained away on this account. After making every allowance, the digestion of the *ragi* straw ration is surprisingly good.

(d) *Comparison of hay results in 1926 and 1927.* Finally the hay results of 1926 and 1927 require notice. It will be recollected that the 1926 ration failed to produce growth, whilst the 1927 ration gave fair growth. The digestion coefficients offer a partial explanation for this difference.

Determinations of the nitrogen balance during digestion experiments.

Nitrogen balances were determined during the digestion experiments of 1926 and 1927. The figures are given in Table XII.

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[1] *Mem. Dept. Agri. India, Chemical Series,* Vol. IX, No. 5.

B

TABLE XII.

Determination of the nitrogen balance in feeding test rations.

	GRM. PER DAY			
	Nitrogen in ration	Nitrogen digested	Nitrogen in urine	Nitrogen assimilated
1926. Hay and concentrate . . .	69·4	38·6	40·4	—1·8
1926. Silage and concentrate . . .	78·1	36·4	30·6	+5·8
1927. Hay and concentrate . . .	74·1	46·2	37·1	+9·1
1927. Silage alone	49·0*	12·8*	11·8	1·0*

* Corrected for a small amount of ammonia present in the silage.

It is not likely that these figures obtained during 6 or 7 days represent accurately the average daily nitrogen assimilation and flesh formation during the entire feeding tests to which they refer, but they should be some guide. Actually, they agree in a general way with the results of the feeding tests. Comparing the two experiments with hay and concentrate, for example, it is seen that in 1927 when growth was good, there was a satisfactory positive nitrogen balance, whilst in 1926 when no live weight increase was obtained, assimilation of nitrogen did not occur. The nitrogen assimilation found with the ration of silage and concentrate in 1926 also corresponds with the fair live weight increase observed in that feeding test. The figures for silage alone in 1927 are highly instructive. Here the protein in the ration was obviously very low, and its digestibility was also low. The animals were only able to absorb small amounts and practically all was excreted. The figures make it very clear why silage alone cannot function as a productive ration.

The relation between food digested and growth.

The average daily food digested can be calculated from the average daily consumption by using the digestion coefficients determined above. Figures obtained in this way are not strictly comparable, because they refer to animals of different live weight. The comparison is somewhat fairer if all results are computed to refer to animals of 1,000 lb. live weight. The accompanying Table gives the results expressed in this form.

Table XIII.

Amount of food digested daily.

	GRAMS FOOD DIGESTED DAILY PER 1,000 LB. LIVE WEIGHT			
	Protein	Total	Nutritive ratio	Live weight increase per 100 days
1926. Hay and concentrate	549·4	3,530	5·9	..
1926. Silage and concentrate	534·1	4,455	7·8	31·0
1927. Hay and concentrate	587·1	4,074	6·4	46·4
1927. Silage alone	251·8	4,170	16·0	..
1928. Low concentrate and silage	495·6	4,922	9·6	61·3
1928. ,, ,, ,, young hay	581·0	5,012	8·2	53·6
1928. ,, ,, ,, mature hay	509·5	4,226	7·9	20·8
1928. ,, ,, ,, *ragi* straw	549·8	4,377	7·6	13·7
1928. High concentrate and silage	608·0	5,197	8·2	79·8
1928. ,, ,, ,, young hay	703·5	4,812	6·4	79·2
1928. ,, ,, ,, mature hay	670·7	4,284	6·0	31·5
1928. ,, ,, ,, *Ragi* straw	689·6	4,549	6·2	46·4

The following points may be noted :—

- (a) Maximum food was digested from the silage and young hay rations of 1928. These rations gave the highest live weight increase and no other ration approaches these either in food digested or in live weight increase. The minimum food was digested in the ration of hay and concentrate of 1926. To this extent the quantity of food digested may be a rough measure of the live weight increase, but it fails to account for the feeding results in the other experiments.
- (b) A high proportion of protein in the digested food has consistently yielded better growth in all cases where similar fodders are compared. This point is well illustrated by the two sets of experiments in 1928. Conversely poor growth is to be expected when the protein digested is low. In this class comes the extreme case of the ration of silage alone (1927) in which failure to grow is clearly due to a deficiency of digestible protein. The Table makes the extent of this deficiency clear. On the whole, the results in this Table are unsatisfactory. The

figures for food digested do not account for the live weight increase except in a very rough way. This conclusion, though it is of a negative kind, is important because it corroborates the Karnal [1] results from which it was likewise concluded that the total food digested is generally not a satisfactory measure for the nutritive value of a ration.

V. Starch equivalent values of feeding test rations.

From the data given in the first part of this paper, it is possible to calculate the starch equivalent values of the rations consumed in each of the feeding tests. Two sets of values, one higher and one lower, will be obtained according as we use American or European standards. But the starch equivalent value of a ration can also be estimated from the live weight increase or nutritive effect. The value so obtained is derived from a source which is independent of food consumption data. It can therefore be used as a check on the values obtained above and should help us to decide whether the American or the European values are more nearly representative for our conditions. These calculations have been carried out for all the spear grass feeding tests at Hosur and the figures obtained are shown in Table XIV.

Table XIV.

Starch equivalent values of rations.

Experiment	Nutritive effect from live weights	Kellners starch equivalent value of ration	Excess of Kellner value over live weight value	American starch equivalent value of ration	Excess of American value over live weight value
1926. Hay and concentrate .	3·40	3·29	—·11	3·62	+·22
Silage and concentrate .	3·95	4·14	+·19	4·44	+·49
1927. Hay and concentrate .	4·00	3·46	—·54	3·86	—·14
Silage alone . .	3·25	2·82	—·43	3·49	+·24
1928. Low concentrate and young hay.	4·97	4·74	—·23	5·70	+·73
Low concentrate and mature hay.	4·21	4·09	—·12	4·58	+·37
Low concentrate and silage.	5·22	4·86	—·36	5·75	+·53
1928. High concentrate and young hay.	5·58	5·20	—·38	6·15	+·57
High concentrate and mature hay.	4·43	4·38	—·05	4·85	+·42
High concentrate and silage.	5·69	5·59	—·10	6·53	+·84
Average .	4·470	4·257	—·213	4·897	+·427

[1] Feeding Experiments at Karnal. *Mem. Dept. Agri. India, Chemical Series*, Vol. X, No. 1.

The evidence of this Table can be stated very briefly :—

(a) In 9 tests out of 10, the European estimate is lower and the American estimate higher than the value obtained from live weight data.

(b) On the average the European figures is 5 per cent. too low and the American figures 10 per cent. too high.

(c) The regularity of the figures adds significance to the differences found.

It would appear from these results that European starch equivalent values can be used with some confidence for Indian foodstuffs. The matter is not so simple however. The Nutrition Section has carried out other experiments (to be reported shortly) on similar lines and it has been found in some cases that the European values are much too low, whilst the higher American values scarcely suffice to account for the observed nutritive effect. That is to say, the nutritive effect is sometimes more and sometimes less than the amount anticipated when rations are judged on the starch equivalent basis. These anomalies may be accounted for in two ways. Either the animals themselves have been more efficient, for unknown reasons, in some tests, or the fodder used in some tests possesses unknown qualities which enhance its value. Which explanation is the true one, it is impossible to say at present, but there is no doubt that a very important line of enquiry has been opened up by these observations. With regard to Hosur it has to be concluded that live weight increase has not been satisfactory. To avoid misunderstanding, the writer would like to say here that he has inspected the cattle on the Hosur Farm frequently and has been struck by the very excellent condition of the live stock generally and by the remarkable growth of the young stock. If this has been accomplished in spite of a relatively poor fodder, the credit to the management is all the greater. The fact remains, nevertheless, that feeding tests have yielded less than the anticipated growth per unit of fodder. It cannot be doubted that this point deserves further study.

Summary.

1. Digestion experiments and feeding tests have been carried out with the following spear grass products :—

Prime hay, mature hay, early cut hay and silage.

2. *Silage as an improved fodder.* The following comparative tests show that silage is eaten more readily and produces more growth than mature hay.

	Food consumption in lb. dry matter per day		
	Fodder	Concentrate	Live weight increase
Silage	9·46	3·45	67·0
Mature hay	9·01	3·34	36·5

c

The better productive power of silage is due partly to better consumption, partly to the high digestibility of the silage carbohydrates.

3. *Early cut hay as an improved fodder.* The Hosur experiments have yielded the following quantitative figures on the advantage of early cutting.

| | FOOD CONSUMPTION IN LB. DRY MATTER PER DAY | | |
	Fodder	Concentrate	Live weight increase
Early cut hay	9·24	3·41	66·5
Mature hay	8·01	3·34	26·5

The better productive power is due partly to better consumption, partly to the high digestibility of the early cut hay. These experiments with early cut hay indicate a line along which the nutrition of cattle in India can be improved.

4. Experiments have indicated that each of these improved fodders, silage and early cut hay, has its own applicability in rationing live stock. The silage is suitable when abundance of concentrate is fed. The early cut hay is useful when economy in concentrate is desired.

5. From extensive digestion trials, the nutritive values of the typical spear grass products have been evaluated and the starch equivalent values of the feeding test rations determined. Thus figures have been obtained by which the Hosur results can be compared with other results obtained by the Nutrition Section. Such a comparison gives an unfavourable impression of the Hosur fodders. Important questions for further investigation have been opened up by the enquiry.

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4

Memoirs of the Department of Agriculture in India

The Nutritive Values of Some Typical Indian Hays

BY

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IMPERIAL INSTITUTE OF AGRICULTURAL RESEARCH, PUSA

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THE NUTRITIVE VALUES OF SOME TYPICAL INDIAN HAYS.

By

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INTRODUCTION.

The Nutrition Section has been carrying out digestion trials of various Indian hays during the past four years. The work has been pressed on systematically, but it has been of an intermittent character, having been done chiefly in periods between other tests, and with any animals that happened to be available at the time. The same animals were not always used. Emphasis is laid on these circumstances here to make it clear that conditions varied in a random manner, and, therefore, the regularities which have been found are signi'cant. The samples tested were obtained from two main sources. The Military Grass Farms provided the largest number. Some of these samples were the ordinary products of the Grass Farms, others were grown at my request. This refers especially to samples in which the effect of the stage of maturity was studied. The other chief source of samples has been the Madras Government Cattle Breeding Farm at Hosur. Hosur has provided samples of the ordinary hay crops, and various samples in which the stages of maturity were studied.

I am greatly indebted to the officers of these two Departments for the valuable samples they have provided to this Section. The result of about three years' labour, proceeding in this manner, is that we possess accurate digestion data for thirty typical hay samples. The figures obtained up to date have been collected together for publication for two reasons. In the first place, there has been a constant demand for information and it seemed that sufficient had been done to warrant a preliminary account of the work. In the second place, the enquiry has reached a phase at which it is necessary to sum up and consider further developments. The analytical work connected with the experiments has been carried out by my staff. I have much pleasure in acknowledging their valuable assistance.

2. THE EXPERIMENTAL DATA.

The results obtained are collected together into three tabular statements. A very important feature of these tables is that the samples have been arranged in the order of protein content. This has been done with a purpose which will become clear later. The order is not chronological.

Table 1 gives the chemical composition of the samples. The distinguishing feature in this table is the figure for protein content. It is seen to range from a maximum of 18·16 per cent. for the richest sample down to 1·98 per cent. for the poorest. It is very likely that the species of grass has a predominant influence here. There are, for example, two samples of Dhub hay and both come very high on the list. The three samples of Kolukattai and the Rhodes grass hays are also high up in the list. These grasses evidently have a relatively high protein content. The very best spear grass sample, on the other hand, is ninth. It must be admitted, however, that the figures are not strictly comparable. Further work must aim at procuring comparable data on this point. Another very important factor governing the protein content is the stage of maturity of the sample. Efforts have been made to collect some information on this point. The following examples are instructive.

1. Spear grass hay from Hosur. The early cut sample is 9th on the list. The same grass cut later is 22nd.

2. Bolarum hay samples 14, 17, and 30 were collected in the same year from the same field at regularly advancing stages of maturity.

3. Kolukattai hay, harvested at three stages, takes up the positions 2, 6, 10 on the list according as maturity advances.

These samples were all cut with the deliberate purpose of determining the effect of advancing maturity. From the samples of known history, it is possible to gain some idea of the state of maturity of the remainder. A reference to the table from this point of view is instructive. The question of maturing, however, deserves a more systematic study, and actually the Nutrition Section organized such an enquiry on a co-operative basis two years ago. Unfortunately the scheme failed, but it will be taken up again in a modified form. There is one more noteworthy regularity to be observed in this table, namely, the association of high soluble ash with high protein content.

Table II gives the digestion coefficients which have been determined by feeding tests for each of the fodders in Table I. The most striking point in this table is the digestibility of protein. It appears that digestibility increases fairly regularly with the amount of protein present. A similar but less regular and less marked increase is apparent in the digestibility of carbohydrates and of organic matter. These facts will be considered more fully from the graphic point of view. The tabular statement is given to record the numerical data obtained in the digestion experiments.

Table III shows the amounts of nutrients provided by each fodder together with starch equivalent values. The figures in this table have been obtained by calculation from the data of Tables I and II. It should be noted here that carbohydrate digestion and the figure for digestible organic matter in this table have been obtained from independent experimental data. Hence it comes about that the sum of protein, ether extract and carbohydrates is not exactly equal to the

figure for digestible organic matter. The two sets of figures are a check on one another.

3. Effect of maturing on the nutritive value of hay.

It was noted above that some tests were made with the definite object of determining the effect of maturity on the nutritive value of hay. To emphasize this important subject, the data relating to maturing are collected together in the accompanying table (the figures are taken from Table III).

Showing the effect of maturing on the nutritive value of hay.

		Digestible nutrients per 100 lb. dry matter			
Source of hay	Stage	Crude protein	Carbo-hydrates	Total Organic matter	Starch equivalent Kellner
Hosur spear grass hay .	Early . . .	2·9	51·4	55·1	38·0
	Ripe . . .	..	50·2	50·1	30·7
Bolarum Rukh hay .	Early . .	2·5	48·7	51·6	32·9
	Prime . .	1·2	49·7	51·7	33·0
	Ripe . . .	..	47·0	46·9	26·0
Kolukattai hay Hosur .	Early . .	10·7	47·6	59·4	44·2
	Prime . .	5·5	49·6	55·8	39·0
	Ripe . . .	3·0	49·5	52·7	34·8

With advancing maturity the amount of digestible protein and the starch equivalent value decrease steadily in every case. The question of maturing is receiving attention at Bangalore.

4. The Graphic representation of digestibility.

The graphic representation of digestibility is given in three charts dealing with protein, carbohydrates and ether extract, respectively.

Chart I. Digestion of protein. This is the most regular set of results obtained. The graph brings out very clearly that there is a close relationship between the amount of protein present in the fodder and the amount digested. It appears that when 16 lb. are present, 12 are digested; when 8 lb. are present 4 are digested. When the quantity present falls to about 2·5 lb., the fodder provides no digestible protein. It is clear that this graph can be used with some confidence for estimating the digestible protein in any hay sample, of which the chemical composition is known. The regularity of the results becomes all the more remarkable when it is recollected that the experiments from which the results have been derived were not carried

out at the same time with the same animals. As already indicated, the digestion trials were done at irregular intervals with the animals that happened to be available at the time. It should be mentioned, however, that the regularity of protein digestion was observed at an early stage of the work and is referred to in my Annual Report for 1926-27. Subsequent tests have amply confirmed the early observations.

Chart II. Digestion of carbohydrates. The chart representing the digestibility of carbohydrates is by no means so regular and in addition to the minor irregularities, there are a few exceptional results which do not conform at all to the general trend of the figures. Nevertheless, it is the writer's opinion that this chart is likely to yield useful information and guidance for future work. In considering the figures in this Table, it has to be borne in mind that we are looking at digestion coefficients of different examples of similar herbage. There should be a regularity in the results and in a general way there is a regularity. The digestibility of the carbohydrates decreases as the protein content of the hay decreases, and there is a tendency to drop rapidly as the protein content becomes low. The group average figures (marked x in graph) make this point more clear. It should be recollected, too, that this is but the skeleton of a scheme. Many minor irregularities would be smoothed out by taking account of differences in species. This will be possible when more data have been procured.

The striking exceptions are quite as noteworthy as the general regularity. It is evident that samples D. M. M. S. & A. are exceptional. The conclusion cannot be avoided that there is something wrong with these samples, and it should be emphasized that the fault has been revealed by applying the graphic method. It is impossible to say what has gone wrong with these samples. The injury may have occurred at harvest time or during growth. At all events, the graph has brought to light the fact that the digestibility of a hay may be depressed at times in an exceptional manner. A very important line of enquiry has been opened up by this observation. Evidently a study should be made of the field conditions which tend to produce such depression. Some steps have been taken to deal with the question. It has been discussed with Officers of the Government Military Dairy and Grass Farm departments and they have agreed to provide samples. More work is desirable however.

Chart III. Digestion of ether extract. This chart shows that there is generally a regularity in the digestion of ether extract and the graph can be used to estimate the digestion of ether extract of any sample of hay, of which the chemical composition is known. There are exceptions again as in the case of carbohydrate digestion, and these exceptions are doubtless significant of peculiar circumstances in the raising and curing of the crop.

5. THE PRACTICAL APPLICATION OF THE GRAPHIC METHOD.

Accurate and reliable digestion data have been obtained for 30 samples of typical hay from different parts of the country. When similar material is met with, the figures given in the tables can be applied with some degree of confidence. But

CHART I.
PROTEIN DIGESTION

PER CENT - PROTEIN IN HAY.

PROTEIN DIGESTED.

CHART II.
CARBOHYDRATE DIGESTION.

ETHER EXTRACT DIGESTED.

PER CENT ETHER EXTRACT IN HAY.

CHART III
ETHER EXTRACT DIGESTION.

B
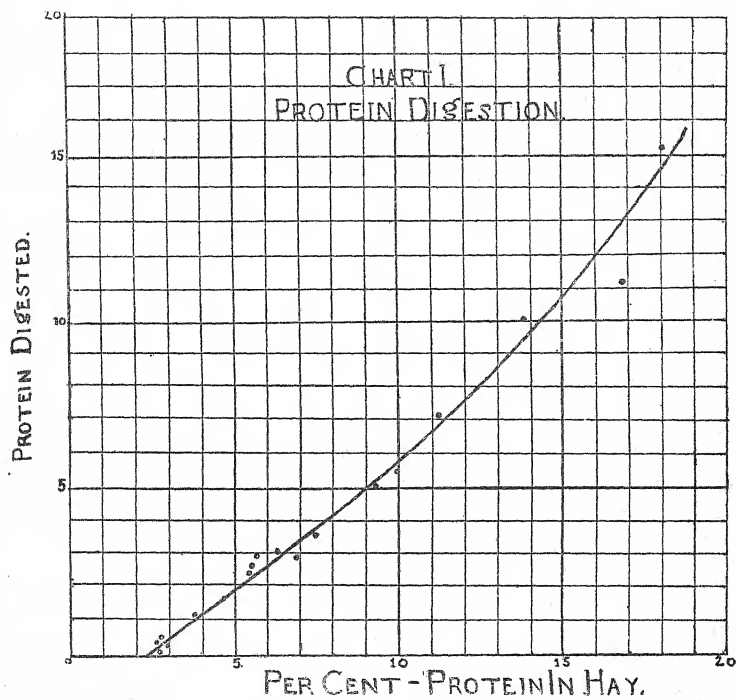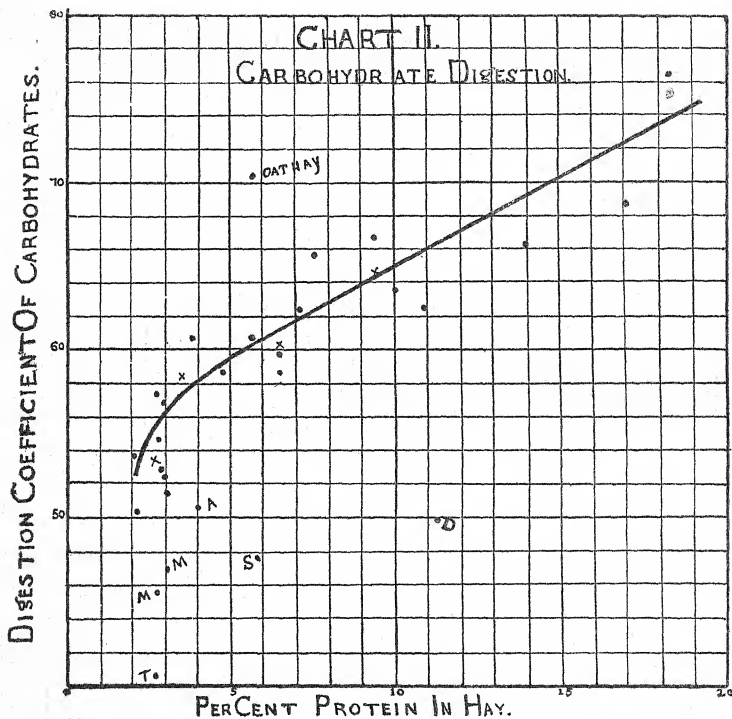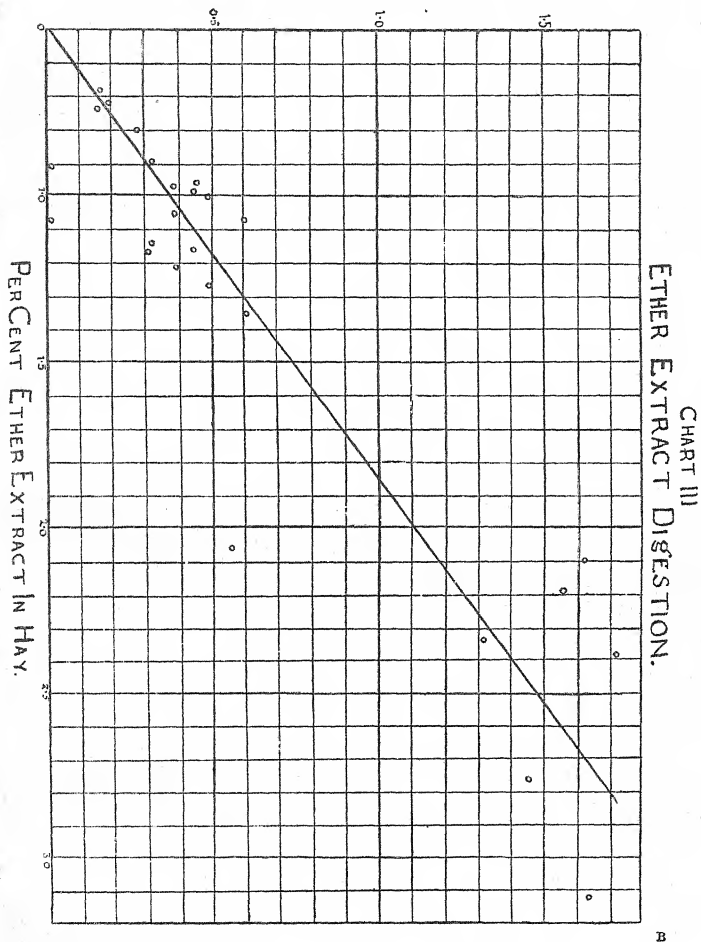
30 samples, though they represent much work, do not cover all the variations that must occur when we recollect that the species of grass, the soil, the climate, the stage of maturity and the harvesting conditions all influence the nutritive value of the product. For the kind of rough estimate with which we must be satisfied for some time to come, the writer believes that the most useful criterion of the quality of a hay is its protein content. For this reason all data in the tabular statements have been arranged according to protein content. Given the chemical composition, with the protein content, a fair estimate can be made of the value of an hay by the following procedure, in which the graphs obtained above are made use of practically.

Example of the utilization of the graphs for estimating the nutritive value of hay.

Given a hay with the following chemical composition on the dry basis :

Protein	6·97
Ether extract	1·35
Carbohydrate	82·40
Ash	9·2
	99·92

we proceed thus :—

1. *Protein.* From Chart I the amount of protein digested can be read directly   3·2
2. *Carbohydrate.* Chart II shows that the digestion coefficient of carbohydrates of hay containing 6·97 per cent. protein is 62 per cent. 62 per cent. of 82·4 is 51·09 = carbohydrate digested   51·1
3. *Ether extract.* From Chart III amount of ether extract digested can be read off directly   ·7

                             Total organic matter digested .   55·1

These figures have been obtained entirely by the use of the three charts. It is true, the figures so obtained may be misleading at times, if they happen to strike a sample in which the digestibility has been appreciably depressed through unknown causes. But exactly the same error would be made if extensive tables of average results were employed to estimate the value of an abnormal product. There is much to be said in favour of the system outlined above, but it can only be accepted as a tentative scheme for the present.

If it is desired to obtain the starch equivalent value of the fodder for which the digestible nutrients were evaluated above, recourse must be had to the writer's starch equivalent graphs, which were published a short time ago in the *Journal of the Central Bureau for Animal Husbandry and Dairying in India*, Vol. III, Part IV. For convenience, these graphs are reproduced in the present paper (Chart IV). In the author's graphic method, starch equivalent values are estimated from the total digestible nutrients or total organic matter digested. In the example above, it was found that the hay yielded 55·1 lb. digestible organic matter per 100 lb. dry substance. The starch equivalent graph (Chart IV) shows that a fodder yielding 55·1 lb. digestible organic matter provides 30 starch equivalents K per 100 lb. dry substance.

STARCH EQUIVALENT.
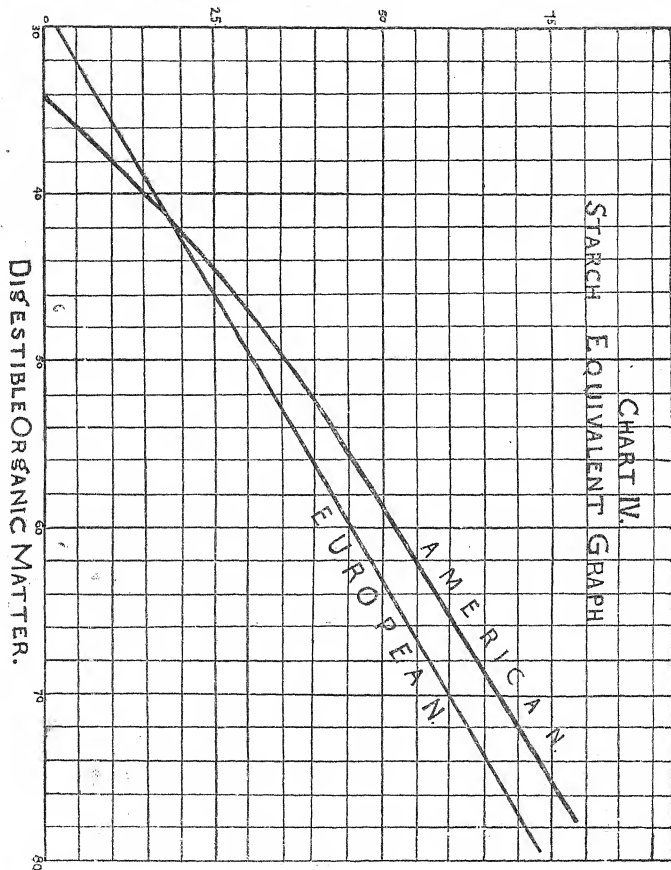
SUMMARY.

1. Full data for the composition and digestibility of 30 typical Indian hays have been procured.

2. The protein content of the samples ranges between 18·16 per cent. and 1·96 per cent. These wide variations are attributable mainly to (a) characteristics of species and (b) to the stage of maturity. More detailed information is needed on both these points.

3. Graphic representation of the data has revealed the fact that the digestibility of all ingredients is related to the protein content of the fodder. This fact can be utilized for estimating roughly the nutritive value of any fodder of which the chemical composition is known. Graphs are provided for this purpose.

4. The graphic method has shown that the digestibility of carbohydrates is at times seriously depressed through unknown causes. This is an important observation which deserves further study.

TABLE I.

Average composition of Indian hays.

Serial No.	Feeding stuffs	Crude protein	Ether extract	Fibre	Nitrogen free extract	Total carbo-hydrates	Ash	Soluble ash
1	Dhub hay, Bangalore, No. I	18·16	2·34	21·1	40·0	61·1	18·46	8·6
2	Kolukattai hay (young), Hosur	16·89	1·14	28·5	40·8	69·3	12·7	8·6
3	Rhodes grass hay, Bangalore	13·96	2·76	29·0	41·3	70·3	13·1	8·9
4	Dhub hay, Bangalore, No. II	11·29	3·12	20·5	50·3	70·8	14·8	5·4
5	Rhodes grass hay, Bangalore	10·77	2·06	25·6	50·4	76·0	11·2	7·0
6	Kolukattai hay (prime), Hosur	10·01	1·12	35·3	42·6	77·9	10·9	7·0
7	Rhodes grass hay (young), Hosur	9·36	1·26	36·2	42·1	78·3	11·1	5·4
8	Rhodes grass hay (prime), Hosur	7·51	1·21	36·6	44·3	80·9	10·4	4·5
9	Spear grass hay (young), Hosur	6·97	1·35	34·5	47·0	82·4	9·2	4·3
10	Kolukattai hay (ripe), Hosur	6·53	0·68	33·3	49·7	83·0	9·9	5·3
11	Rhodes grass hay (ripe), Hosur	6·36	1·16	39·3	44·1	83·4	9·1	3·9
12	Seguri hay	5·84	1·00	38·4	44·8	83·2	9·9	1·9
13	Oat hay, Lahore	5·63	2·39	22·6	61·9	84·5	7·5	4·4
14	Bolarum hay (young)	5·56	1·16	36·0	44·3	80·3	10·0	4·5
15	Anjan hay, Meerut	4·73	0·87	30·4	53·8	84·2	10·3	4·9
16	Ahmednagar hay	3·85	0·98	30·9	42·0	78·9	16·3	2·3
17	Bolarum hay (prime)	3·81	1·05	36·9	45·7	82·6	12·5	3·5
18	Meerut Farm hay of 1927, Over matured.	3·04	0·78	40·3	46·4	86·7	9·5	3·1
19	Meerut Farm hay of 1927, Over matured.	2·69	0·72	40·8	45·8	86·6	9·8	2·8
20	Meerut Farm hay of 1926, Over matured.	2·97	2·19	27·9	48·1	76·0	18·9	3·7
21	Meerut Farm hay of 1927, Over matured.	2·94	0·72	40·6	45·3	85·9	10·4	3·1
22	Mature spear grass hay, Hosur	2·86	0·74	37·7	50·7	88·4	8·0	3·3
23	Meerut Farm hay of 1928, Over matured.	2·84	1·07	36·7	47·3	84·0	12·1	2·7
24	Rukh hay (ripe), Bolarum	2·74	0·96	31·4	51·8	83·2	13·1	2·8
25	Talegaon hay	2·72	0·97	37·5	43·1	80·6	16·7	2·3
26	Meerut Farm hay of 1926, Over matured.	2·66	1·08	36·3	42·6	78·9	10·3	3·0
27	Meerut Farm hay of 1926, Over matured.	2·55	0·91	36·3	41·4	77·7	18·9	3·2
28	Mature spear grass hay, Hosur	2·52	0·90	42·7	46·0	88·7	7·8	3·3
29	Rukh hay (prime), Bolarum	2·10	2·09	26·9	51·6	78·5	17·4	3·4
30	Bolarum hay (ripe)	1·98	0·86	38·3	49·4	87·7	9·4	1·8

TABLE II.

Average digestibility of Indian hays.

Serial No.	Feeding stuffs	Crude protein	Ether extract	Total carbo-hydrates	Total organic matter	Soluble ash	No. of tests
1	Dhub hay, Bangalore, No. I .	83·95	56·04	76·48	77·57	79·92	1
2	Kolukattai hay (young), Hosur .	66·14	27·70	63·65	″67·98	82·75	2
3	Rhodes grass hay, Bangalore .	72·44	52·57	66·23	67·19	82·8	1
4	Dhub hay, Bangalore, No. II .	63·81	52·36	50·04	52·19	61·0	1
5	Rhodes grass hay, Bangalore .	66·52	26·70	62·49	62·58	77·13	2
6	Kolukattai hay (prime), Hosur .	54·86	41·49	63·59	62·63	75·76	2
7	Rhodes grass hay (young), Hosur .	49·51	32·51	65·74	64·23	57·47	2
8	Rhodes grass hay (prime), Hosur .	54·80	38·85	66·66	65·31	60·56	2
9	Spear grass hay (young), Hosur .	42·15	44·21	62·34	60·63	47·6	6
10	Kolukattai hay (ripe), Hosur .	46·58	22·98	59·58	58·69	58·25	2
11	Rhodes grass hay (ripe), Hosur .	48·85	38·36	58·64	59·11	26·62	2
12	Seguri hay	51·77	48·91	47·60	47·86	32·3	3
13	Oat hay, Lahore . . .	45·97	71·87	70·33	69·20	74·67	2
14	Bolarum hay (young) . .	44·81	27·72	60·70	59·27	54·2	3
15	Anjan hay, Meerut . . .	35·02	30·03	58·74	57·46	69·32	2
16	Ahmednagar hay . . .	31·16	46·16	50·51	50·99	36·57	2
17	Bolarum hay (prime) . .	31·71	37·53	60·61	59·03	58·50	3
18	Meerut Farm hay, 1927, Over matured	8·86	53·93	50·55	49·45	52·24	3
19	Meerut Farm hay, 1927, Over matured	8·06	35·27	51·16	49·64	39·97	3
20	Meerut Farm hay, 1926, Over matured	16·46	71·18	46·63	48·60	58·61	2
21	Meerut Farm hay, 1927, Over matured	8·60	23·35	52·25	50·47	50·5	3
22	Spear grass hay (mature), Hosur .	··	21·80	56·79	54·53	35·4	6
23	Meerut Farm hay, 1928, Over matured	10·06	56·24	52·75	51·19	53·85	3
24	Rukh hay (ripe), Bolarum . .	14·33	47·72	54·58	53·42	61·1	2
25	Talegaon hay	1·9	40·16	40·45	39·37	5·2	2
26	Meerut Farm hay, 1926, Over matured	3·16	··	45·49	43·35	60·7	2
27	Meerut Farm hay, 1926, Over matured	3·16	··	45·49	43·35	60·7	2
28	Spear grass hay (mature), Hosur .	··	36·21	57·34	55·09	50·14	3
29	Rukh hay (prime), Bolarum .	··	77·32	50·26	49·86	56·91	2
30	Bolarum hay (ripe) . . .	··	32·66	53·53	51·80	40·73	3

TABLE III.

Digestible nutrients in 100 lb. dry matter of Indian hays.

Serial No.	Feeding stuffs	Crude protein	Ether extract	Total carbo-hydrates	Total organic matter	Soluble ash	STARCH EQUIVALENT	
							American	European
1	Dhub hay, Bangalore, No. I .	15·26	1·31	46·69	63·25	6·87	57·0	50·0
2	Kolukattai hay (young), Hosur	10·67	0·32	47·55	59·35	7·37	51·1	44·2
3	Rhodes grass hay, Bangalore .	10·07	1·45	46·90	58·42	7·33	49·6	42·8
4	Dhub hay, Bangalore, No. II	7·24	1·63	35·56	44·48	3·29	24·6	22·6
5	Rhodes grass hay, Bangalore .	7·16	0·55	47·84	55·55	5·40	45·2	38·6
6	Kolukattai hay (prime), Hosur	5·49	0·46	49·58	55·80	5·30	45·6	39·0
7	Rhodes grass hay (young), Hosur	5·13	0·49	52·20	58·07	3·10	49·2	42·3
8	Rhodes grass hay (prime), Hosur	3·72	0·39	53·17	57·55	2·73	48·4	41·6
9	Spear grass hay (young), Hosur	2·94	0·60	51·40	55·10	2·05	44·5	38·0
10	Kolukattai hay (ripe), Hosur .	2·97	0·16	49·46	52·86	3·09	40·7	34·8
11	Rhodes grass hay (ripe), Hosur	3·11	0·45	49·91	53·74	1·04	42·0	36·0
12	Seguri hay	3·02	0·49	39·62	43·11	0·61	21·5	20·5
13	Oat hay, Lahore . . .	2·58	1·72	59·73	64·03	3·29	58·0	51·6
14	Bolarum hay (young) . .	2·49	0·31	48·74	51·58	2·33	38·5	32·9
15	Anjan hay, Meerut . . .	1·65	0·24	49·66	51·55	3·40	38·5	32·0
16	Ahmednagar hay . . .	1·20	0·45	39·83	42·67	0·84	20·7	19·0
17	Bolarum hay (prime) . .	1·21	0·39	49·86	51·66	2·05	38·7	33·0
18	Meerut Farm hay of 1927, Over matured.	0·26	0·26	44·40	44·93	1·12	25·4	23·1
19	Meerut Farm hay of 1926, Over matured.	0·49	1·56	35·75	37·80	2·17	9·4	12·8
20	Meerut Farm hay of 1927, Over matured.	0·25	0·17	44·37	45·20	1·57	26·1	23·6
21	Spear grass hay (mature), Hosur .	—1·00	0·16	59·19	50·14	1·17	35·8	30·7
22	Meerut Farm hay of 1928, Over matured.	0·54	0·60	46·44	45·01	1·59	25·6	23·3
23	Rukh hay (ripe), Bolarum . .	0·39	0·46	45·57	46·42	1·46	28·5	25·3
24	Talegaon hay	0·05	0·39	32·60	33·18	0·12	(—1·5)	6·1
25	Meerut Farm hay (late harvest), 1926.	0·08	..	35·89	35·97	1·82	5·1	10·2
26	Meerut Farm hay (late harvest), 1926.	0·08	..	35·35	35·43	1·94	3·6	9·3
27	Spear grass hay (mature), Hosur .	—0·04	0·32	50·86	50·08	1·65	35·8	30·7
28	Rukh hay (prime), Bolarum .	..	1·62	39·58	41·20	1·94	17·3	17·7
29	Bolarum hay (ripe) . .	—0·6	0·28	46·96	46·91	0·73	27·6	26·0

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Memoirs of the Department of Agriculture in India

The Nutritive Value of Gram Husk

BY

F. J. WARTH, M.Sc.,

Physiological Chemist, Imperial Department of Agriculture

AND

LAL CHAND SIKKA, B.Sc. (Agri.),

Punjab Department of Agriculture

THE NUTRITIVE VALUE OF GRAM HUSK.

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OBJECT OF EXPERIMENT.

The object of the experiment was to study the function and nutritive value of gram husk. In India various husks are used as cattle food. Gram husk is one of the commonest and most widely used. As there are no data available regarding the nutritive value of these husks, a study of the subject seemed to be of practical importance.

PROCEDURE.

Three groups of calves were selected for the test. Group A received ground-nut cake as the sole concentrate. Group B received the same allowance of cake to which was added an equal weight of wheat bran. Group C received the same ration as group B, except that gram husk replaced the wheat bran. Previous experiments carried out by the Nutrition Section having shown that increments of concentrate should not be made simply proportionate to live weight, the following schedule of concentrate rationing was adopted.

Live weight lb.	Ration of ground-nut cake, grams.
200	250
300	341
400	400
500	455

Fodder was provided in the form of fresh Guinea grass and dry *ragi* straw. The former was fed at the rate of 1·5 kilos per 100 lb. live weight, the latter was fed

(85)

ad. lib, but the daily consumption by each animal was determined. From a larger number of animals originally chosen, many had to be eliminated, 21 being finally retained. The preliminary feeding consisted of an initial period during which all received the same food, and a transition period during which the animals were brought on to the test rations. The feeding test lasted 12 weeks, at the end of which a digestion trial was conducted with 3 selected animals from each group.

The senior author wishes to acknowledge gratefully the help given by his staff in carrying out all the analyses connected with this enquiry.

2. Food Consumption.

(a) Consumption of Guinea grass.

The fresh Guinea grass was fed in proportion to live weight at the rate of 1·5 kilos per 100 lb. live weight. The fodder provided was generally completely eaten up. However, the moisture content of the material underwent a periodical change, increasing steadily for a time and then decreasing again. In consequence, the amount of dry matter actually fed varied. The weekly group average consumption of Guinea grass dry matter is shown in chart 1. The marked sag in the graphs indicates the period during which the dry matter content of the herbage fell. It will be noticed that group A ate distinctly less Guinea grass than groups B and C. This is due to the fact that Guinea grass was fed in proportion to live weight ; and as this group made less live weight increase, it received slightly less fodder.

(b) Consumption of ragi straw.

Ragi straw was fed *ad. lib* to all animals and they were encouraged to eat as much as possible. Chart II represents *ragi* straw consumption. A steady increase in *ragi* straw consumption is noticeable throughout the experiment. There are two factors to account for this increase. In the first place, the animals were being trained steadily to eat more. In the second place, the decreased consumption of Guinea grass (explained above) gave more room for *ragi* straw consumption. This graph is interesting in showing clearly that group A consumed more *ragi* straw than groups B and C. This fact will be referred to again.

(c) Total roughage consumption.

Chart III representing total roughage consumption is instructive in showing that although *ragi* straw consumption increased, when Guinea grass decreased. The increase in the former was not sufficient to compensate for the decrease in the latter. There is still a sag in the consumption curve though it is less marked here than in the Guinea grass curve. Towards the end of the test, larger amounts of both fodders were eaten. This chart showing total roughage consumption will be referred to again when live weight increase is considered.

Chart I — Guinea Grass Consumption
(weeks / Dry matter in kilos)

Chart IV — Live Weights
(weeks / Live weight)

Chart III — Total Roughage Consumption
(weeks / Dry matter in kilos)

Chart II — Raga Grass Consumption
(weeks / Dry matter in kilos)
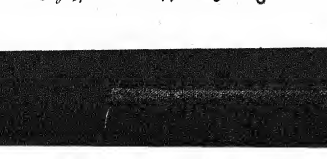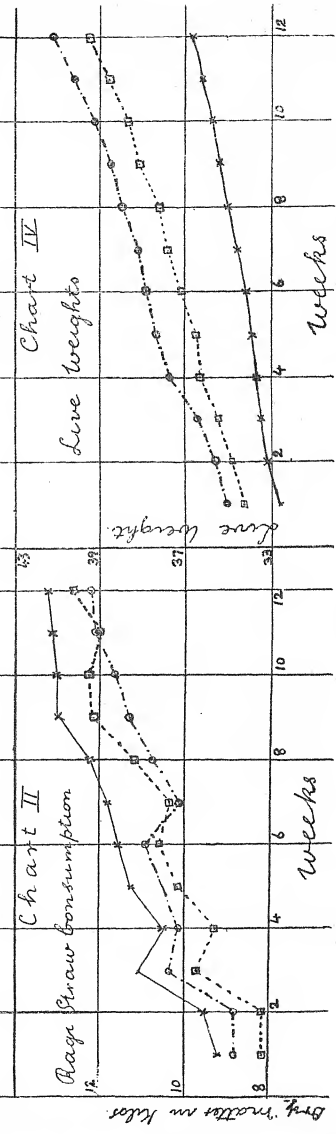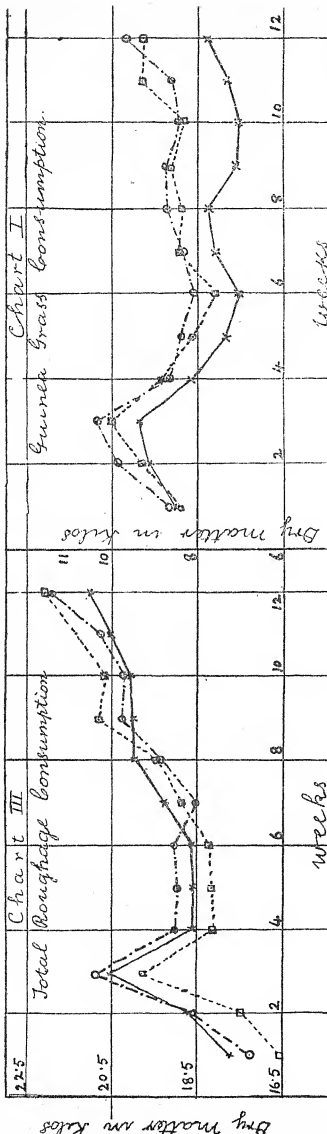
(d) *Concentrate consumption.*

Concentrate was fed strictly according to the schedule and was completely consumed.

(e) *Average food consumption.*

The group average food consumption in kilos dry matter for the entire feeding period of 84 days is given in Table I.

TABLE I.

Group average food consumption in kilos dry matter.

Group			TOTAL CONSUMPTION IN KILOS DRY MATTER					Total	Total roughage
			Ragi straw	*Guinea grass*	*Ground-nut cake*	*Wheat bran*	*Gram husk*		
Actual.	A .	.	139·8	94·2	57·5	. .	. .	291·5	234·0
	B .	.	126·7	105·8	60·2	57·6	. .	350·3	232·4
	C .	.	125·6	103·5	59·3	. .	59·5	347·9	229·1
Per 880 lb. Live weight.	A .	.	149·4	100·6	61·5	. .	. .	311·5	250·0
	B .	.	121·7	101·6	57·8	55·3	. .	336·4	223·3
	C .	.	123·3	101·6	58·2	. .	58·5	341·6	224·9

It appears from these figures that the actual consumption of total roughage is about the same for the 3 groups. Hence the total food consumption is less for group A than for groups B and C simply by the amount of extra concentrate given to the 2 latter groups. In this type of ration the addition of bran or gram husk induces so much additional food consumption without affecting the consumption of roughage. The feeding results show clearly that gram husk functions in this respect exactly like wheat bran, weight for weight. This is an important observation. The results may be looked at in another way. The groups were equal in live weight at the commencement, but groups B and C increased more rapidly, hence the food consumed by these groups really refers to animals of slightly greater average live weight. From the actual food consumption of each group, the consumption of an animal having the average live weight of the herd can be computed. The computed values are shown in the lower half of the above table.

The latter figures indicate that the animals of group A consumed relatively more roughage than groups B and C. This was an evident attempt by the animals of group A to make up for the deficiency of concentrate by increasing their roughage

consumption. The figures show that in the type of ration tested a shortage of concentrate could not be effectively balanced by increased roughage consumption.

(f) *Food consumption per 1,000 lb. live weight.*

The consumption per 1,000 lb. live weight works out to 21·5, 23·2 and 23·5 lb. dry matter for groups A, B, C. Judged by other tests carried out at Bangalore, the consumption by groups B and C may be considered moderately satisfactory. For group A, the consumption is somewhat low.

3. LIVE WEIGHT DATA.

(a) *Live weight data of individuals.*

Table II shows the total food consumption and total live weight increase of each individual.

TABLE II.

Relation between total food consumption and live weight increase.

Group A			Group B			Group C		
Average live weight	Total food	Live weight increase	Average live weight	Total food	Live weight increase	Average live weight	Total food	Live weight increase
313·8	298·6	58·5	405·7	395·7	80·6	385·9	336·5	85·5
333·8	311·7	52·5	375·0	348·8	76·5	359·1	335·6	65·8
307·4	290·0	50·8	315·8	297·2	74·9	292·6	324·6	79·8
230·7	190·7	17·2	272·6	258·6	62·0	276·6	266·2	64·2
438·0	318·2	36·9	458·4	383·1	64·3	453·0	389·0	73·2
474·2	336·1	34·2	519·4	389·4	77·1	505·4	397·3	92·0
391·6	295·1	18·2	422·5	378·1	63·9	436·5	336·	58·5
355·6	291·5	38·3	395·6	350·2	70·5	387·0	347·9	74·1

The details in this Table are given to show the kind of evidence we have for preparing group average results. The food consumption varies naturally with the size of the animal, but even when full allowance is made for size, it appears that the relationship between food consumption and live weight increase is not close. Individual efficiency has had a considerable effect in producing live weight increase. This possibility was given due weight when the animals were originally grouped

for the test. In every case three well balanced animals were taken and one put into each group. The effect of this initial selection can be clearly seen in the above table. For example, the fifth animal in each group was intended to form a good trio for comparison. These are the smallest animals in their respective groups, they ate least, and they made least growth in their groups. The seventh animals were matched similarly. They were larger but all somewhat unthrifty. These three animals also gave very low results for their respective groups, as the Table above makes clear. Thus we feel that though there are evident individual differences in efficiency, the differences have been successfully distributed between the three groups. The efficiency of the animals having been balanced, the group average results can be accepted as a fair measure of the relative efficiencies of the rations.

(b) Group average live weight data.

The group average live weight changes are shown graphically in chart 4. Each point on these graphs represents the average of seven consecutive days' weighings of 7 animals. The chart brings out two points clearly. Firstly groups B and C behaved identically. From start to finish, they increased at an equal rate. Secondly, group A increased quite regularly but at a distinctly slower rate.

One peculiarity in the rate of live weight increase is worth a remark. It will be noticed that the slopes of the graphs are practically constant except for a short space about one-third of the way through the experiment. At this stage live weight increase was slightly retarded in all the groups. A reference to chart 3 (total roughage consumption) shows that this retardation in growth occurred when roughage consumption fell off.

We can conclude from these graphs that the nutritive effect of rations B and C was, as nearly as we can judge, identical, while ration A was distinctly less effective. The actual live weight increases for the 3 groups A, B and C during the test were 33·3, 70·5 and 74·1 lb. respectively. That is to say, additions of wheat bran and gram husk have produced almost identical increments above the basal ration and the increment has been considerable. The data relating to live weight may be stated in the following condensed form.

TABLE III.

Summary of live weight data.

Group	Average live weight lb.	Average daily increase lb.
A	355·6	0·45
B	395·6	0·84
C	387·0	0·88

To sum up, the evidence of the feeding test, it has been found that " in rations such as were used, gram husk improves the ration much in the same way as wheat bran and approximately to the same extent. Both substances improve the consistency and palatability of the ration and both cause an approximately equal increase in food consumption and in live weight".

4. DIGESTION EXPERIMENTS.

(a) *Digestibility of gram husk.*

The only available information on the digestibility of gram husk is from unpublished figures obtained by the Nutrition Section at Bangalore. These are given in the accompanying Table together with corresponding figures for wheat bran.

TABLE IV.

Constituents of gram husk and wheat bran per 100 lb. dry matter.

	IN FOOD			AMOUNT DIGESTED			Kellner's starch equivalent*
	Protein	Ether extract	Carbohydrates	Protein	Ether extract	Carbohydrates	
Gram husk .	5·96	·77	85·35	·00	·54	59·43	42·4*
Wheat bran .	15·90	3·85	73·37	12·40	2·62	47·64	50·6*

*Graphic values.

Two points in this Table require special notice. Firstly, the digestion experiments carried out by the Nutrition Section show that gram husk has an appreciable starch equivalent value. The value appears to be distinctly lower than that of wheat bran, while the feeding test indicated approximately equal values. However, in the feeding test wheat bran and gram husk formed relatively small proportions of the total ration and the figures for live weight increase give but an approximate measure of the value of the total ration. From such data it is not possible to gauge accurately the value of a fraction of the ration. The feeding test, therefore, corroborates, as well as could be expected, the value found by digestion experiments. Secondly, it has to be noted that gram husk provides no digestible protein whatever. In this respect it differs widely from wheat bran. Therefore where there is any question of adequacy of protein or efficiency of protein in a ration, these two foods are not comparable. Wheat bran would probably be very helpful, while gram husk would be useless. To avoid misunderstandings, it is necessary to lay emphasis on this important distinction. Furthermore, it is not sufficient to say that gram husk provides no digestible protein. In the digestion experiment quoted above, it was found that gram husk causes a small but distinct depression in the digestion of protein from mixed rations.

(b) Digestibility of the experimental rations.

At the end of the feeding test three animals from each group were retained on their respective rations for a further period of two weeks. During this time a digestion trial of the feeding test mixed ration was carried out. As the three animals on each ration behaved absolutely alike, it is unnecessary to give the results separately for each. The average consumption and average digestion attained by each group are given in the accompanying Table.

TABLE V.

Nutrients digested from feeding test rations (grams per day).

		Dry matter	Crude protein	Ether extract	Nitrogen free extract	Fibre	Total carbo-hydrates	Nutritive ratio
Group A.	Total nutrients in food.	3937·6	514·89	116·14	1644·3	1248·5	2892·8	··
	Nutrients digested.	2137·4	360·63	82·04	891·3	659·4	1550·7	4·8
	Computed digestion.	··	361·05	79·12	904·1	752·3	1656·4	
	Difference between computed and actual.	··	—0·42	+2·92	—12·8	—92·9	—105·7	··
Group B.	Total nutrients in food.	4901·7	671·69	154·67	2150·2	1425·1	3575·3	··
	Nutrients digested.	2790·7	484·21	111·31	1245·4	766·3	2011·7	4·7
	Computed digestion.	··	488·66	106·13	1276·9	843·2	2120·1	
	Difference between computed and actual.	··	—4·45	+5·18	—31·5	—76·9	—108·4	··
Group C.	Total nutrients in food.	4774·9	584·82	131·86	1951·3	1631·1	3582·4	··
	Nutrients digested.	2669·3	374·37	91·58	1087·1	949·2	2036·3	6·0
	Computed digestion.	··	381·33	94·24	1144·8	1018·9	2163·7	
	Difference between computed and actual.	··	—6·96	—2·66	—57·7	—69·7	—127·4	··

For purposes of comparison, the theoretical computed digestion is added. These figures were calculated from digestion coefficients which have been determined by the Nutrition Section at Bangalore in other experiments. The coefficients are recorded in a subsequent table. It will be noticed that the agreement between the theoretical and actual results is close. The protein digestion comes up to the theoretical amount almost completely, the most serious departure (Group C) being 1·8 per cent. of the total quantity, and part of this deficiency must be attributed to the depressing effect of gram husk on protein digestion. There is a small but very regular deficiency in the actual digestion of carbohydrates. This was to be expected, because we are dealing with young animals whose capacity for digesting coarse fodder has not attained its full power. Taking this fact into consideration, the digestion attained by our test animals is remarkably good. The most important point to observe in this table is the amount of protein digested. Rations A and C provide practically identical amounts, namely, 360 to 370 grams per day. Ration B, the wheat bran ration, provides very much more, namely, 480 grams. Again emphasis must be laid on this marked distinction of the wheat bran ration. The figures show once more that gram husk, useful though it may be, cannot function as wheat bran in this respect.

The nutritive ratios of the 3 rations are of considerable interest too. Rations A and B give very similar and moderately narrow ratios, while ration C has a wider ratio. The last figure reflects the higher food consumption brought about by gram husk feeding without a corresponding increase in digestible protein.

(c) *Percentage of nitrogen in food and faeces.*

The figures for percentage nitrogen in the food and faeces show the facts from another point of view. In the accompanying table the average of the sum total nitrogen percentage in food and faeces is given.

TABLE VI.

Percentage nitrogen in food and faeces.

	A	B	C
Percentage nitrogen in food	2·09	2·19	1·96
,, ,, ,, faeces	1·37	1·42	1·62

In rations A and B the amount of nitrogen in faeces is proportional to the amount in food. In ration C the proportion of faecal nitrogen is much higher, showing again the presence of undigested nitrogen from the gram husk. The

digestion experiments have added considerably to the information gained from the feeding tests. The facts established may be more fully stated thus :—

Gram husk is a moderately digestible food, its starch equivalent value being 4/5 that of wheat bran. It provides carbo-hydrates but no protein. Its chief effect is that of an appetiser. It improves the consistency and palatability of rations and leads to increased food consumption thereby generally widening the nutritive ratio. In these respects it must be frequently very useful and beneficial in the rations of cows, as long as the bulk of the ration is not large otherwise. It must be recognized from the data obtained that gram husk is not a concentrated food.

Nitrogen balance experiment.

To determine the retention of nitrogen, a short test was carried out with one animal from each group at the end of the feeding test. The results are recorded in the accompanying table together with relevant data.

TABLE VII.

Nitrogen balances.

—	Group A	Group B	Group C
Nitrogen digested daily grm.	53·3	80·6	61·6
„ excreted in urine grm.	46·6	51·8	51·0
Nitrogen assimilated daily	6·7	28·8	10·6
Kellner's starch equivalent available for live weight* increase.	0·83	1·44	1·19
American starch equivalent available for live weight* increase.	1·44	2·56	2·09

* Note these starch equivalent figures refer to the period of the nitrogen balance experiment only.

The figures indicate that high assimilation was taking place on ration B and low assimilation on rations A and C. The high assimilation is associated with a high figure for nitrogen digested and low assimilation with low figures for nitrogen digested. But in the cases of low assimilation the amounts digested were sufficient to permit of much higher assimilation. Clearly assimilation is at fault in rations A and C. Looking at the starch equivalent figures, it will be seen that assimilation has undoubtedly been influenced in this test by the amount of food consumed. The animal on ration B had the greatest excess of starch equivalent available for live

weight increase. It would therefore be able to assimilate most nitrogen. In this way the starch equivalent figures certainly help to account for a part of the differences in nitrogen assimilation but they can scarcely account for the whole difference. There is a possibility that the question of physiological efficiency of proteins is involved in this result. It has to be recollected that the nitrogen balance test was carried out at the end of a feeding trial when physiological deficiencies in the protein of rations A and C may have become perceptible. The efficacy of wheat bran from this point of view deserves study.

5. Starch Equivalent Values.

Composition and Digestibility of Food-stuffs.

The accompanying table gives the average composition of the food-stuffs used in the feeding tests. The material collected for chemical analysis was thoroughly representative, sampling having been done every day during the test.

Table VIII.

Chemical composition of food-stuffs.

	PERCENTAGE COMPOSITION					DIGESTION COEFFICIENTS				STARCH EQUIVALENT		
	Crude Protein	Ash	Ether extract	Nitro-gen free extract	Crude fibre	Crude protein	Ether extract	Nitro-gen free extract	Crude fibre	Kell-ner	Gra-phic	Ame-rican
Guinea grass .	6·06	14·06	1·72	36·40	41·14	52·43	42·87	51·38	57·73	23·4	24·5	28·8
Gram husk .	5·94	6·51	1·32	35·41	50·83	..	100·00	71·33	65·72	44·8	43·5	50·5
Wheat bran .	17·04	4·61	3·75	63·38	11·22	78·00	68·00	72·00	31·00	52·0	52·5	60·5
Ground-nut cake	50·45	3·85	9·85	33·10	6·61	89·64	97·00	51·44	9·81	81·5	81·5	98·4
Ragi straw .	4·16	7·91	0·96	49·80	38·17	..	..	61·01	70·15	35·0	37·0	44·0

The analyses indicate that the wheat bran was a particularly good sample especially with respect to protein ; gram husk, ground-nut cake and *ragi* straw were good ; guinea grass was satisfactory in all respects except that the ash content was somewhat high. In estimating the nutritive values of all these foods, Bangalore digestion coefficiencies have been used except in the case of wheat bran for which American values have been employed.

The senior author has recently called attention to certain difficulties in estimating the net energy values of Indian Fodders (*Journ, Central Bureau for Animal Husbandry and Dairying in India*, Vol. III, Pt. IV, p. 119). While the present doubt exists, estimates are made according to both European and American authorities. The results of these estimates are given in the Table above, all being expressed in starch equivalent units.

It will be noticed that the estimates by the Bangalore graphic method agree very well with Kellner's values. The American values are decidedly higher. In the case of roughages the difference ranges between 5 and 8 starch equivalent. With groundnut cake there is a very serious divergence between European and American estimates. We are not in a position to comment on this matter at present. Combining the starch equivalent values found above with the data relating to food consumption, we can calculate the starch equivalent values of the rations actually consumed by the 3 groups during the feeding test. According as American or European values are used for the individual food-stuffs, higher or lower values will be obtained for the rations. Both sets of values are shown in the Table.

The Nutritive effect of the rations.

The nutritive effect is seen from and measured by the live weight increase. In the accompanying table the figures in lines A and B are estimates of the nutritive effect of the 3 rations. The figures in line A were obtained by assuming that 1 lb. live weight increase=1·5 starch equivalent. This seems to us a fair allowance. It is highly improbable that the animals received less than this amount of net energy from the ration. The higher values in line B were obtained by assuming that 1 lb. live weight increase =2·0 starch equivalent. This is perhaps a somewhat high value for the class of animal tested.

TABLE IX.

Nutritive effect and values of rations in starch equivalent per day.

Group	Ration A	Ration B	Ration C	Average	
A	3·81	4·29	4·38	4·16	Values obtained from live weight data.
B	4·05	4·69	4·81	4·52	
C	3·30	3·73	3·68	3·57	Values obtained from food consumption data.
D	4·06	4·53	4·46	4·35	
E	3·79	4·28	4·20	4·09	

For comparison with the figures derived from live weight data, we have figures derived from food consumption data. The latter are given in lines C, D and E, line C corresponding to the lower European values (Kellner's), line D corresponding to the higher American values. These two different values have been fully explained above. Line E has been calculated by using the high American values for all food-stuffs except groundnut cake for which the lower value accepted in

Europe has been used. The data collected together in this Table appear to show conclusively that European starch equivalent values (Line C) are too low for the food-stuffs employed by us. In no case do they come up to the lowest estimate that can be allowed for the observed nutritive effect (Line A). Using American values for all food-stuffs except groundnut cake (Line E), figures are arrived at which agree with the lowest estimate for the observed nutritive effect. From this we conclude that the nutritive effect of the rations is most nearly represented by assigning American values to all the food-stuffs except groundnut cake. On the value of groundnut cake the evidence is not clear.

Evidence from other experiments.

To obtain further evidence the senior author has examined the data of two long period feeding tests carried out at Bangalore. In the first experiment (1) mature bullocks were used. It is assumed that 2·7 starch equivalent should be allowed per lb. live weight increase for these animals. In the second experiment (2) calves weighing 360 to 370 lb. were used. For these animals 1·5 starch equivalent have been allowed per lb. live weight increase. Treating the data as above, the following results are obtained.

TABLE X.

Nutritive effect and starch equivalent of rations, Bangalore.

| | | | CALVES (2) | |
		Mature bullocks (1)	Guinea grass + Concentrate	Silage + Concentrate
Values from live weight data . . .	..	5·27	4·12	4·35
Values from food consumption data . .	Kellner .	4·60	3·56	3·75
	Graphic .	4·26	..	..
	American .	5·04	3·78	4·20

In all these experiments the Kellner's starch equivalent values seem to be very low compared with the live weight estimates. For the bullock experiment it is true a high figure has been assigned to the starch equivalent required for 1 lb. live weight increase. For the class of animal considered, the figure 2·7 is not excessive ; but even if only 2·0 starch equivalent were allowed, the live weight data

would still indicate a nutritive effect of 5·11 starch equivalent instead of 5·27 given in the table above. The conclusion is inevitable that Kellner's starch equivalent values underestimate the nutritive effect in this experiment. The data from Bangalore are summarized in the following statement.

TABLE XI.

Summary of Bangalore results.

Experiment	Number of animals in test	Nutritive effect from live weight	STARCH EQUIVALENT VALUES OF RATIONS			
			KELLNER'S VALUE		AMERICAN VALUE	
			Starch equivalent in ration	Excess over column 3	Starch equivalent in ration	Excess over column 3
1	21	4·16	3·68	—0·48	4·35	+0·19
2	6	5·27	4·60	—0·67	5·04	—0·23
3	8	4·12	3·56	—0·56	3·78	—0·34
4	8	4·35	3·75	—0·60	4·20	—0·15
	Average	4·48	3·90	—0·58	4·34	—0·13

It would appear from these figures that at Bangalore the Kellner's values are much too low, while the American values barely suffice to account for the nutritive effect. It remains, finally, to compare the Bangalore and Hosur results. In the accompanying Table the Hosur figures are taken from *Mem. Dept. Agri. India, Chemical Series*, Vol. XI, No. 3.

TABLE XII.

Comparison of Bangalore and Hosur results.

	No. of animals tested	Kellner's value	American value
Bangalore	43	—·58	—·13
Hosur	80	—·21	+·47

That is to say, at Hosur the nutritive effect appeared to be well below American values and slightly above Kellner's values. At Bangalore the nutritive effect appears to have been much greater, being slightly above the American estimates for food values. Although the differences are considerable, the writer is not prepared to affirm that they are significant. It is a difficult matter to obtain a correct appreciation of the real nutritive effect of a ration. If the data obtained are not significant, they are certainly very suggestive and provide a means for critically examining and comparing results of feeding trials.

SUMMARY.

1. A comparative feeding test showed that, in the type of ration used, gram husk affects the ration much in the same way as wheat bran. Both substances improve the consistency and palatability of the ration and both bring about approximately parallel increases in food consumption and live weight.

2. Digestion experiments show that gram husk possesses a distinctly lower starch equivalent value than wheat bran. A marked distinction between wheat bran and gram husk is that the latter provides no digestible protein whatever.

3. Gram husk cannot be considered a concentrated food but it possess undeniably valuable properties, some of which are clearly defined by the experimental results obtained.

4. Comparing starch equivalent values of the mixed rations with the observed nutritive effect of these rations, it is concluded that the relatively higher American values correspond more closely to the effect observed in this experiment.

5. Other experiments carried out by the Nutrition Section at Bangalore are quoted to show that American values frequently give close approximations to the observed nutritive effect.

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Memoirs of the Department of Agriculture in India

Studies in Soil Colloids, Part III

Flocculation of soil colloids

BY

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STUDIES IN SOIL COLLOIDS, PART III.

FLOCCULATION OF SOIL COLLOIDS.

BY

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Clarification of turbid water with alum has long been known to people in certain parts of India where there is scarcity of water.

As far back as 1868 Skey [1] published a short note on the coagulation and precipitation of clay by neutral salts generally:

He arrived at the following important conclusions :—

(1) The power of clarification of the various salts studied were in the following order :—H_2SO_4 > Lime > Ca and Ba Chloride > Chloride and Sulphate of Na.

(2) Upon washing the clay precipitate, it reacquires its tendency of permanent diffusion.

(3) The quantity of clay matters present appears of secondary importance ; complete precipitation having nearer relationship to the degree of dilution allowed to the salt employed.

In 1871 Schloesing [2] confirmed some of Skey's results and further showed that chloride, nitrate, sulphate bicarbonate and hydrate of calcium have apparently an equal effect. He explained the formation of deltas as being due to coagulation of the suspension by the electrolytes in the salt water. Schloesing was probably the first to point out the gradually increasing effect on flocculation as the concentration of flocculating agent is increased; for instance, he found that $CaCl_2$ $\frac{1}{1000}$ of the liquid, precipitated immediately, $\frac{1}{5000}$ after some minutes, $\frac{1}{50000}$ in two or three days.

---

[1] Skey. *Chemical News*, Vol. 17, p. 160 (1868).
[2] Schloesing. *J. C. S.*, **24**, p. 750 (1871).

In 1907 Hall and Morrison [1] published a detailed study of the phenomenon of flocculation in clays. They worked mostly with Kaolin and Natrolite. They did not attempt to put forward any theory and in fact showed that none of the theories advanced in the case of flocculation of other colloidal solutions fitted in with their observations and measurements.

In 1916 Wolkoff [2], summarizing the most important literature on the subject, gave some results of flocculation experiments on clays from different origins and showed that the flocculating efficiency of different electrolytes depends on the nature of the clay, a fact hitherto not recognized by previous workers.

In 1918 Pickering [3] studied the flocculation of Kaolin and clay among other substances and called attention to the large increase in the volume occupied by the particles as evidenced by the volume of the deposit, eventually obtained on subsidence. He ascribed flocculation to a combination of the flocculant with Kaolin.

From 1920 onwards Comber [4] published a series of three papers in which he developed the theory that soil particles consist of a core with a gel coating ; the former imparting to them the character of a suspensoid and the latter emulsoid. The essential difference between particles of various sizes being determined by the ratio of emulsoid surface to the core of the particles. In clay the surface, in silt the core, dominates the system. This theory accounted for the abnormal behaviour of clays, the flocculation of which by Ca ions is facilitated by the presence of hydroxyl ions. He also showed that the absorptive power of a soil for lime must be satisfied before the latter can cause abnormal flocculation of clay.

Of special interest in this connection is the work of Gedroiz.[5] He arrived at the following important conclusions :—

    (1) The flocculating power of acids is roughly inversely proportional to their dissociation constant. Completely dissociated acids like HCl, HNO_3 and H_2SO_4 being required in equivalent amounts.

    (2) Except Rb, & Ag. all the chlorides of monovalent ions were equally potent in their flocculating power.

    (3) The flocculating action of the chlorides of bivalent ions like Mg, Ca, Sr, Cd, Ba, Mn, Ni and Co was equal but considerably higher than monovalent ions. With trivalent ions like Al and Fe the action was still stronger than mono- or divalent ions.

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[1] Hall & Morrison.   *Jour. Agr. Sci*, **2**, p. 244 (1916).

[2] Wolkoff.   *Soil Sci.*, **1**, p. 585 (1916).

[3] Pickering.   *Proc. Roy. Soc.*, A. Vol. XCIV, p. 315 (1918).

[4] Comber.   *Jour. Agr. Sci.*, **10**, 1920 ; 11 (1921) ; 12 (1922).

[5] Gedroiz.   Communication 24 from the Bureau of Agr. & Soil Sci. of the Scientific Committee of the main department of Landorg & Agri., Petrograd, 1915.

Up till now little attempt has been made to study the flocculation of soils with due regard to the phenomenon of base exchange. Without a knowledge of the nature and extent of the cationic exchange likely to be effected in a given soil on the addition of a given electrolyte, it would be impossible to build up a comprehensive theory of the stability and flocculation of soil colloids.

In line with the evidence brought forth in support of the general theory of soil acidity and base exchange, the terminology adopted in the previous paper of this series will be maintained. So that a completely "unsatured" clay will be called clayic acid and its neutralization products with bases as clayates. The use of terminology that emphasizes the chemical nature of the various reactions that can be brought about with clays is to be infinitely preferred as providing a proper perspective in their understanding. The following therefore is a study of the flocculation of soil suspensions containing clayic acid and various clayates.

EXPERIMENTAL.

It has been shown by several workers that the process of flocculation is not abrupt but proceeds with a gradually increasing intensity as the concentration of the flocculant is increased above a certain minimum. The complete course of flocculation, however, need not be followed, but only those concentrations of various electrolytes can be determined which produce the same change in the solution within a given time. These flocculation values (F. V.) can be used for comparing the relative action of various electrolytes on a given suspension. Three criterions were used for fixing the flocculation values : i.e., the appearance of a clear layer of supernatant liquid on (A) leaving the suspension for 24 hours ; (B) leaving the suspension for 10 minutes ; (C) Centrifuging for 3 minutes at 1500 r. p. m. These will be referred to as A, B, or C method in the text.

FLOCCULATION OF CLAYIC ACID SUSPENSIONS WITH HYDROXIDES.

(1) Effect of concentration of the suspension on F. V.

Particles of 0·001 mm. limiting diameter were separated from a heavy clay soil (Dharwar, Bombay) after acid treatment when all the clayates had been converted into clayic acid. Increasing amounts of water were added to 10 c.c. portions of a 4 per cent. suspension and the minimum concentration of alkali required to produce flocculation within 10 minutes noted down in each case. In Table I are given F. V.'s for the hydroxides of Ca, Sr, and Ba, at various concentrations of the

suspension. The results plotted in Fig. 1 show that there is a logarithmic relationship between concentration of suspension and F. V.; and that almost equivalent amounts of Ca, Sr and Ba hydroxides are required to flocculate a soil.

Effect of Conc. of suspension on F.V. for hydroxides of Ca, Sr,&Ba.
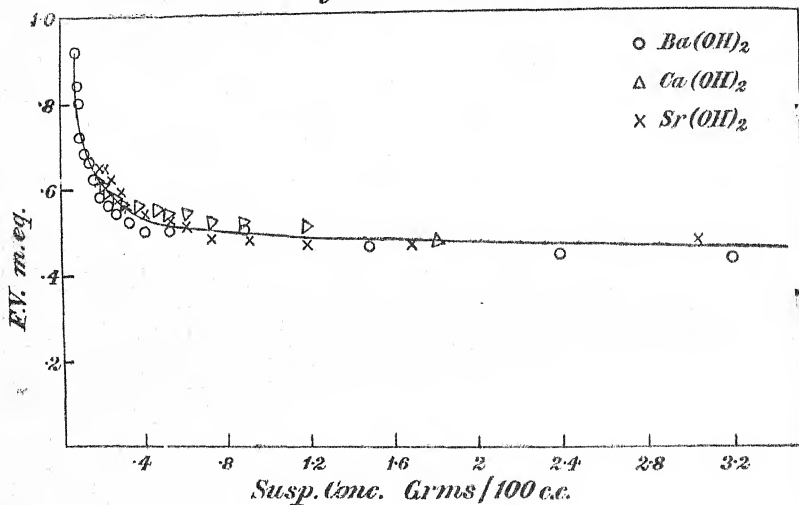
Fig. 1.

TABLE I.

Effect of concentration of suspension on F. V. for hydroxides of Ca, Sr, and Ba.

Ba (OH)$_2$		Sr (OH)$_2$		Ca (OH)$_2$	
Susp. Conc. grams-100 c.c.	F. V. m. eq.	Susp. Conc. grams-100 c.c.	F. V. m. eq.	Susp. Conc. grams-100 c.c.	F. V. m. eq.
3·246 . . .	·436	3·057	·475	. .	. .
2·430 . . .	·441	1·726	·475	1·793	·4725
1·478 . . .	·46	1·199	·475	1·204	·509
·9009 . . .	·50	·9179	·482	·912	·524
·5445 . . .	·50	·7448	·462	·7408	·524
·4180 . . .	·50	·6258	·508	·6204	·537
·3375 . . .	·52	·5378	·524	·534	·546
·2834 . . .	·54	·423	·541	·4684	·553
·2440 . . .	·56	·348	·558	·3781	·561
·2140 . . .	·58	·2953	·59	·3169	·569
·1901 . . .	·62	·2559	·624	·2725	·585
·1710 . . .	·62	·2261	·656	·2329	·593
·1564 . . .	·66	·2028	·656	·2129	609
·1331 . . .	·68	. .	. .	·1917	·625
·1156 . . .	·72	. .	. .	. .	. .
·1020 . . .	·80	. .	. .	. .	. .
·0915 . . .	·84	. .	. .	. .	. .
·0826 . . .	·92	. .	. .	. .	. .

It might be noted that the minimum F. V. is only a little higher than the amount of alkali required for neutralizing the clayie acid up to the H$_2$ limit (formation of mono-hydrogen acid saloid*).

This point is examined in greater detail in Table II wherein are given the flocculation values for a number of acid treated soils containing different amounts of clayie acids ; 5 grams of soil being suspended in 100 c.c. of water. It will be seen that F. V. in each case is approximately the same as the base neutralizing

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*Clayie acid is regarded as tribasic acid. See Part 1 of these studies.

power up to H_2 limit (equivalent to CO_2 evolved on heating with $BaCO_3$[1]), showing thereby that very little $Ba(OH)_2$ is required to flocculate a soil after its base neutralizing power up to H_2 limit has been satisfied.

TABLE II.

Showing the base neutralizing power of acid treated soils (up to H_2 limit) and F. V. for $Ba(OH)_2$.

Soil	Base neutralizing power up to H_2	F. V.
	M. eq. for 5 grams	M. eq. for 5 grams
A. T. 1	1·215	1·25
A. T. 2	0·825	0·95
A. T. 3	1·08	1·03
A. T. 4	1·485	1·97
A. T. 5	1·395	1·65
A. T. 6	0·795	0·76
A. T. 7	1·46	1·34
A. T. 8	1·305	1·34

The fact that the base neutralizing power of a soil must be satisfied before lime or $Ba(OH)_2$ can flocculate it, has been noticed both by Gedroiz as well as Comber (loc. cit.) and seems to show that flocculation of a soil suspension is not brought about by the adsorption of this or that ion, but by its being in solution. It is also well known in the domain of colloid chemistry that substances that are adsorbed in equivalent amounts by a given colloid may have widely differing flocculation values. Fruendlich has explained this on the supposition that strongly adsorbed ions are adsorbed at a much lower equilibrium concentration and hence that neutralization of the charge takes place at a much lower concentration; concluding thereby that more strongly adsorbed ion has a smaller F. V. This view is not compatible with the fact that NaOH is equally well " adsorbed " by the soil if not better than $Ba(OH)_2$ and yet it is required in a very much higher concentration to produce flocculation than the latter.

---

[1] Cf. Part I of these studies.

We have seen that Ba clayate must be formed first when $Ba(OH)_2$ is added to clayic acid soil suspension ; and it is only the amount of $Ba(OH)_2$ over and above that amount, which produces flocculation. It was of interest to see how this value would be modified if a part of the clayic acid was neutralized with NaOH. For this purpose Akola soil was first converted to the acidoid state and then neutralized (pH 7) with a mixture of $Ba(OH)_2$ and NaOH, thus were obtained series of soil suspensions containing increasing ratios of Na and Ba clayates. Flocculation with $Ba(OH)_2$ was studied on them. Results given in Table III and plotted in Fig. 2 show that as the percentage of exchangeable Na increases, F. V. for $Ba(OH)_2$ increases correspondingly.

Flocculation of Soils.
With increasing amounts of Na
in the Exchange complex with Ba (OH)₂
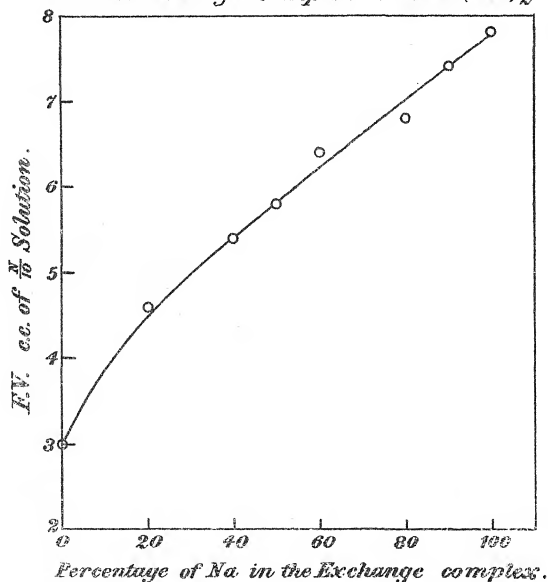
Fig. 2.

TABLE III.

Flocculation of soil suspension containing increasing amounts of exchangeable Na in the clay, with Ba(OH)$_2$.

Percentage of exchangeable Na on total amount of bases.	F. V. c.c.'s of N/10 Ba(OH)$_2$ per 25 c.c. of 2·5% suspension.
0	1·5
20	2·3
40	2·7
50	2·9
60	3·2
80	3·4
90	3·7
100	3·9

The above results can be easily interpreted if we remember that part of Ba is exchanged by Na and thus rendered inactive as it were, because F. V. for Na salts is much higher than Ba salts.

Experiments with a 2 per cent. Na clayate soil suspension confirmed the results obtained with clayic acid soil suspension that an equivalent amount of the hydroxides of Ca, Ba, Sr is required for causing flocculation in a given suspension.

It is worth noting that the reaction between the soil and the hydroxides of divalent ions proceeds rather slowly especially near the neutralization point (H$_2$); hence larger amounts of these hydroxides are required if the suspension is left for some time with the flocculant than otherwise.

II. FLOCCULATION WITH ACIDS.

Flocculation with acids is not of great interest except when soil colloid is amphoteric, *i.e.*, having high sesquioxide content and isoelectric point, cf. Mattson[1], because

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[1] Mattson, S. Anionic and Cationic adsorption by Soil Colloidal material of varying SiO$_2$/Al$_2$O$_3$ + Fe$_2$O$_3$ ratio. *Proc. Int. Cong. Soil. Sci.*, II Commission, p. 199, Washington (1928).

of the inevitable side reactions due to the dissolution of Al which acts as a powerful flocculant. Indeed, it is doubtful if the acids have got very great flocculation power, it seems more likely that the flocculating action of H ions falls more in line with the weak action of monovalent ions.

The dissolution of Al by acids is a slow process and therefore very much smaller quantity of acid is required to produce flocculation if the suspension is left with flocculant for sometime than otherwise. This is shown in Table IV wherein are recorded flocculation values for H_2SO_4 for the same suspension at different intervals of time. To 25 c.c. portions of 2 per cent. sodium clayate soil suspension increasing amounts of $\frac{N}{20}$ H_2SO_4 were added and the tubes centrifuged (C method) after different intervals of time and flocculation noted.

TABLE IV.

Effect of time of contact on flocculation by H_2SO_4.

Time interval (hours)	F. V. (c.c.'s of $\frac{N}{20}$ H_2SO_4)
0·1	15
0·5	13·5
1·0	10·0
2·0	9·0
3·0	8
4·0	8
19	7
24	6·5
47	6·5

III. FLOCCULATION WITH NEUTRAL SALTS.

It might be stated at the outset that the influence of electro-negative ion in chlorides, nitrates and sulphates was found to be negligible. This conclusion has been arrived at by other workers and needs no amplification.

Flocculation with NaCl of soil suspensions containing one kind of exchangeable ion was studied. The results given in Table V leave no doubt as to the fact that the nature of the exchangeable ion is of prime importance in the study of flocculation of soil colloids.

TABLE V.

Flocculation of soil suspensions containing one kind of exchangeable ion with NaCl.

Soil suspension exchangeable ion	F. V. for NaCl M. eq. per 100 c.c.
Na	12·28
Mg	1·575
Ca	0·566
Sr	0·3846
Ba	0·3846
H	0·1935
Al	0·00385
Fe	0·00458

There are probably two causes operating in the phenomenon of flocculation as influenced by the nature of the exchangeable ion.

1. The higher the valency of exchangeable ion, the lesser the ionisation and stability of the suspension and hence the lesser the F. V. for a given electrolyte.

2. A part of the Na ions in the NaCl are exchanged by an ion of a higher valency (which is more potent in producing flocculation) in all suspensions except No. 1.

The effect of exchangeable sodium is also seen in a striking manner in the case of a series of soil suspensions containing increasing amounts of Na, the rest being Ba, described before (Table III).

The results, given in Table VI and plotted in Fig. 3, bring out the following points.
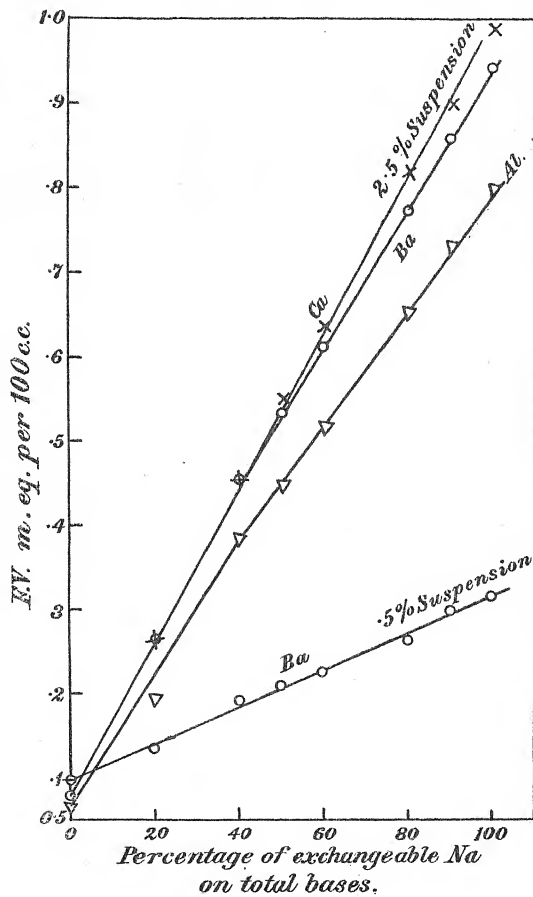
Fig. 3.

(1) Difference between F. V. for Ba and Ca ions increases as the amount of exchangeable Na increases in the clay.

(2) Flocculation values for Al ions are not vastly different from those for Ca and Ba ions, as would have been expected, from the results given in Table V. This is most probably due to the formation of Na aluminates at higher pH values which behave like other Na salts as regards their flocculating power ; or it may be due to precipitation of a part of Al as $Al(OH)_3$.

(3) Mg ions are not so powerful flocculating agents as Ba and Ca. This conclusion is at variance with that of Gedroiz who found Mg ions not much different from other divalent ions.

(4) In conformity with the conclusions arrived at by other workers, Na ions are found to be the weakest flocculating agents, also they show a great difference between F. V. for partial and complete flocculation and where the amount of exchangeable Na is large, it is not possible to locate the latter value with exactitude, as the whole mass gelatinises. Perhaps measurements of viscosity changes would give a more exact F. V.

(5) The difference between F. V. for Ba ions with 0·5 per cent. and 2·5 per cent. suspension systematically increases as the amount of exchangeable Na in the clay increases, reaching a maximum when the suspension contains no other exchangeable ion except Na.

TABLE VI.

Flocculation of soil suspension containing increasing amounts of exchangeable Na in the clay with various ions (chlorides).

Percentage of exchangeable Na on total bases	F. V. (M. EQ. PER 100 C.C.)						
	Ba		Ca	Mg.	Al	Na (2·5 per cent. Susp.)	
	2·5 per cent. Susp.	0·5 per cent. Susp.	2·5 per cent. Susp.	2·5 per cent. Susp.	2·5 per cent. Susp.	Partial Floc.	Complete Floc.
0 . .	·0787	·0980	·0787	0·6238	·0593	·3968	·9802
20 . .	·2650	·1362	·2650	1·065	·1923	1·923	2·830
40 . .	·4546	·1923	·4547	1·473	·3875	4·546	7·628
50 . .	·5357	·2107	·5516	1·805	·4546	5·357	8·334
60 . .	·614	·2290	·6445	1·9182	·5198	6·140	9·680
80 . .	·777	·2651	·8194	2·1995	·6598	6·898	Indefinite
90 . .	·861	3007	·9016	2·323	·7338	9·68	Indefinite
100 . .	·9412	·3184	·9936	2·428	·8054	12·12	Indefinite

The effect of increasing concentration of Na clayate soil suspension on F. V. for Ca ions was examined in greater details in the case of the last named suspension that contained 100 per cent. exchangeable Na. The results recorded in Table VII and plotted in Fig. 4 show that the effect is regular and systematic.

Flocculation of Na clayate Suspension with Ca SO₄, at various concentrations.
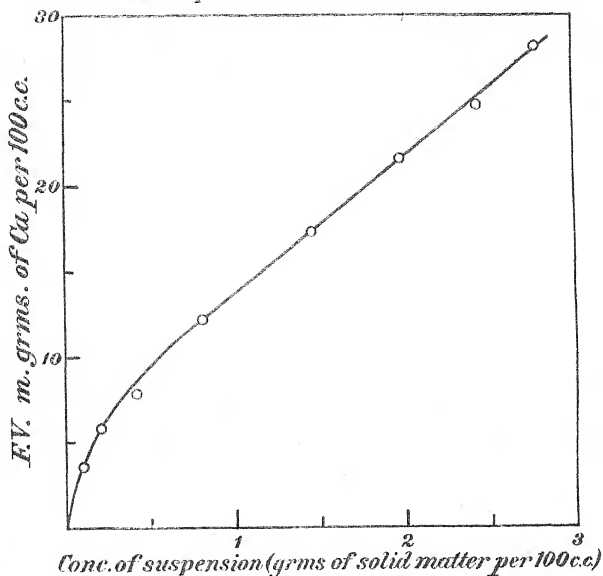
Fig. 4.

TABLE VII.

Flocculation of Na clayate suspension with $CaSO_4$ at various concentrations.

Conc. of susp. at the flocculation point. gram-100 c.c.						F. V. Milli-grams Ca ions per 100 c.c.
0·118	·	·	·	·	·	3·59
0·227	·	·	·	·	·	5·76
0·439	·	·	·	·	··	7·78
0·806	·	·	·	·	·	12·37
1·453	·	·	·	·	·	17·37
1·973	·	·	·	·	·	21·69
2·439	·	·	·	·	·	24·74
2·778	·	·	·	·	·	28·18

The above results, along with those recorded in Table VI, have a significance of practical value. They show, for instance, that the concentration of gypsum needed for flocculating a deflocculated soil containing only divalent exchangeable ions is 1·574 milli-grams of Ca per 100 c.c. (apart from the fact that this concentration rises to 19·08 milli-grams per 100 c.c. when the divalent ion is replaced by Na) shows that lime cannot be a permanent cure for the deflocculated condition of a soil; because since all lime must get converted into $CaCO_3$, a saturated solution of which contains only 0·72 milli-grams of Ca per 100 c.c. (or little more if some $CaHCO_3$ is formed), the deflocculated condition is bound to return. It also explains the frequently observed fact that even calcareous soils containing as much as 16 per cent. $CaCO_3$ can deteriorate in fertility by the accumulation of exchangeable Na and the only remedy for such soils would be the application of gypsum. The above remarks, of course, do not apply to soils rich both in exchangeable Na as well as exchangeable H (degraded alkali soils) which must have lime for neutralizing the acidity.

All the experiments reported so far were done with suspension neutralized up to H_1 stage; (di-hydrogen acid saloid) since such a soil can take up another equivalent of alkali for converting it into the monohydrogen acid saloid[1]; it was of interest to see the effect on the flocculating power of a salt when a di-hydrogen acid clayate is gradually neutralized with NaOH.

Increasing amounts of $\frac{N}{10}$ NaOH were added to 50 c.c. portions of 1 per cent. suspension of a di-hydrogen Ba clayate soil and F. V. for $BaCl_2$ determined in each case.

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[1] Puri. Studies in Soil Colloids, part I. Base Exchange and Soil Acidity. *Mem. Dept. Agri, India, Chem. Ser.,* Vol. XI, No. 1.

The results given in Table VIII and plotted in Fig 5 show that F. V. for Ba Cl$_2$ increases systematically with increasing amount of NaOH added to the suspension reaches a maximum and then decreases systematically ; but the interesting thing is that the maximum occurs at the point where the formation of monohydrogen saloid is complete (neutralization up to H$_2$ stage). It is clear that on the ascending portion of the curve the presence of hydroxyl ions* retards flocculation and on the descending portion in the same clay the flocculation is facilitated by the presence of

Effect of increasing amount of Na OH on the F.V. for Ba Cl$_2$.
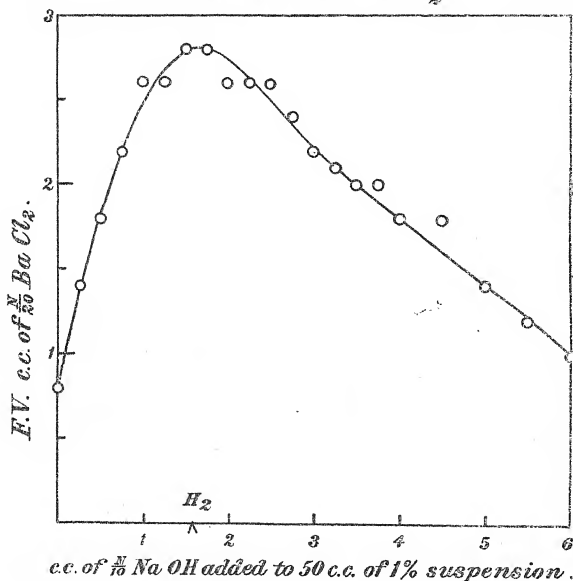
FIG. 5.

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* The effect is not due to the absence of the products of exchange acidity, because the soil being already neutralized with Ba(OH)$_2$ up to H$_1$ stage, no exchange acidity could be produced by the addition of BaCl$_2$. See part I of these studies.

B

hydroxyl ions. It seems, therefore, that Comber's theory of "emulsoid" coating on the clay particles would be unnecessary to account for the anomalous behaviour of clay, because before the reality of any such anomaly could be established, a knowledge of the nature and amount of exchangeable ions would be necessary.

TABLE VIII.

Effect of increasing amounts of NaOH on the F. V. for $BaCl_2$.

C.c. of $N/10$ NaOH added to 50 c.c. of 1 per cent. suspension	C.c. of $N/50$ $BaCl_2$ required to flocculate
0	0·8
·25	1·4
·5	1·8
·75	2·2
1·0	2·6
1·25	2·6
1·5	2·8
1·75	2·8
2·0	2·6
2·25	2·6
2·5	2·6
2·75	2·4
3·0	2·2
3·25	2·4
3·50	2·0
3·75	2·0
4·0	1·8
4·5	1·8
5·0	1·4
5·5	1·2
6·0	1·0

The effect of addition of NaOH to Al clayate suspension on the F. V. for $Al_2 Cl_6$ is shown in Fig. 6. Unlike the flocculation of Ba clayate with $BaCl_2$, there is no maximum for F. V. up to the concentration studied, the reason is that Al ions being amphoteric in character cease to exist in alkaline medium. This is marked by a slight change of direction in the F. V. curve.

Effect of addition of NaOH to
Al Clayate suspension on the F.V. for $Al_2 Cl_6$.
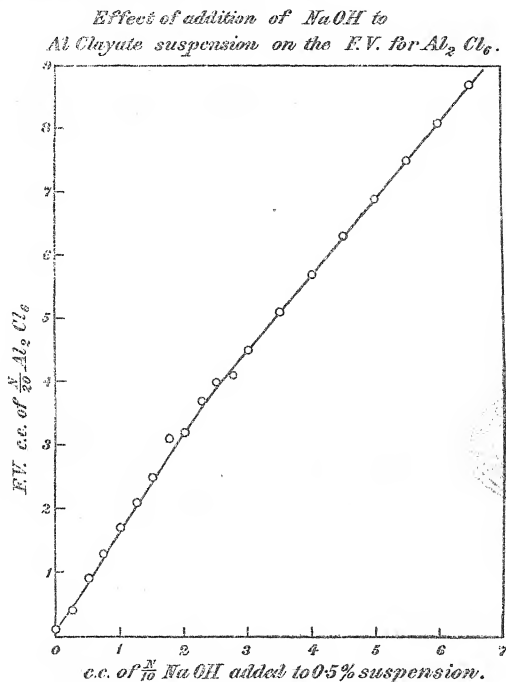
Fig. 6.

Some attention has been directed recently towards the importance of pH control in all studies of soil flocculation. While this is likely to lead to useful results, the fundamental cause of the importance of pH values in this phenomenon must not

be lost sight of. It must be recognized that variations in pH are themselves due to changes in the state of saturation of clay. The soil can be brought to any desired pH value both by $Ba(OH)_2$ as well as NaOH and yet the effect on flocculation is totally different in the two cases.

SUMMARY.

(1) Flocculation of clay suspensions has been studied, with particular reference to the nature of the exchangeable ion in soil colloids.

(2) It appears that the nature of the replaceable base in a soil has a profound influence on the flocculation values for different ions.

(3) Of special interest are the results with soils containing increasing amounts of exchangeable sodium in them; which show that as sodium increases, the flocculation value for calcium ions increases correspondingly. The importance of these results in the practical reclamation of alkali soils is pointed out.

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Studies in Soil Colloids, Part IV

Methods of estimating soil colloids

BY

AMAR NATH PURI, Ph.D., M.Sc., A.I.C.

Late Physical Chemist, Imperial Institute of Agricultural Research, Pusa

STUDIES IN SOIL COLLOIDS, PART IV.

METHODS OF ESTIMATING SOIL COLLOIDS.

BY

AMAR NATH PURI, Ph.D., M.Sc., A.I.C.,

Late Physical Chemist, Imperial Institute of Agricultural Research, Pusa.

A good deal of attention has been directed during the past few years towards methods of estimating the amount of colloidal materials in soils. The literature on the subject up to 1924 has been summarized by Gile et al.[1] The methods so far suggested can be grouped as follows :—

    1. Mechanical analysis methods.
    2. Adsorption methods :
        (*a*) Water vapour adsorption.
        (*b*) Dye adsorption.
        (*c*) NH_3 adsorption.
    3. Heat of wetting method.
    4. Shrinkage method.
    5. Chemical method.

It appears that three factors are involved in any consideration of soil colloidal behaviour, namely :—

    (*a*) The size of the particles.
    (*b*) The surface development, and
    (*c*) Surface reactivity.

The above factors are neither identical nor entirely inter-dependent as far as soil colloids are concerned. The fact that no satisfactory method has yet been discovered of estimating soil colloids, is chiefly due to the failure in attaching a precise meaning to the term " soil colloid " ; and it would require no elaborate argument to prove that unless that is done it would be futile to prescribe any method for measuring it. Recently Joseph has drawn attention to this point and has put forward the workable suggestion that for the present the inorganic soil colloids may be taken as synonymous with the clay content[2].

The utilitarians urge that any estimation of soil colloids must be correlated with the behaviour of the soil in the field. Even on this basis it would be difficult

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[1] Gile, Middleton, Robinson and Anderson. *U. S. Dept. Agri. Bull.* No. 1193 (1924).
[2] Joseph. *Soil Sci.*, 24, p. 271 (1927).

to choose between the size, the surface, or reactivity of particles. Each one of these factors plays its own part, and there is no reason to reject one in favour of the others. The ideal method therefore would be the one that combined all these factors and indicated the resultant effect, what we are really after is not an estimation of soil colloids but a measurement of colloidal behaviour, for such behaviour is admittedly of importance in any correlation between soil conditions and plant growth.

A method of estimating soil colloids can be either absolute or relative. If the former, it can be used for soil characterisation and as such should be uninfluenced by the natural changes ordinarily going on in the soil during comparatively short intervals of time, if the latter, then a knowledge of the effect of natural causes on its magnitude is of prime importance before it could be made to serve any useful purpose, practical or theoretical.

As far as we know, the newer methods of mechanical analysis (such as the International method, or the author's NaCl—NaOH method[1]) could be depended upon to give absolute values for clay ; values that would not change from month to month or even year to year.

If we, therefore, adopt Joseph's suggestion that clay may be considered synonymous with colloidal matter, the question becomes simple. However, a method may be absolute and yet arbitrary and this is the argument in the hands of the advocates of other methods. They point out that it may be possible to determine the percentage of certain arbitrarily fixed size with exactitude, but mere size takes no account of the surface development nor it gives any indication as to whether the latter is smooth or rugged ; crystalline or amorphous, inactive or active. Adsorption and heat of wetting methods, on the other hand, would presumably give a truer measure of surface reactivity and consequently a better estimate of colloidal behaviour of soils than the mechanical analysis. The fact that they often give too high a value for colloidal content and include perhaps a portion of silt along with clay as colloidal, is an argument rather in favour of the " adsorption " and heat of wetting methods than against them ; for a colloid does not lose its colloidal behaviour when spread over an inert surface ; in other words, a surface can just as well be colloidal as particles. However, it remains to be seen to what extent adsorption and heat of wetting methods are absolute. In other words, how far the values obtained are influenced by such factors as the nature of the exchangeable ion, presence or absence of soluble or insoluble salts, etc.

The effect of exchangeable bases in the soil on moisture absorption has been studied by Thomas[2] ; on heat of wetting by Pate[3] and on dye adsorption by Gedroiz.[4] The effect on the ammonia adsorption has not yet been studied.

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[1] Puri, A. N. A New Method of Dispersing Soils for Mechanical Analysis. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. X, No. 8 (1929).
[2] Thomas, M. D. *Soil Sci.*, 25, p. 485 (1928).
[3] Pate, W. W. *Soil Sci.*, 20, p. 329 (1925).
[4] Gedroiz, K. K. *Zhur. Opit. Agron.*, 15, p. 181 (1914).

The following conclusions have been arrived at :—

(1) Moisture absorption (especially at higher humidities), heat of wetting and dye absorption are greatly influenced by the nature of the replaceable base in the mineral complex.

(2) On the whole, a soil saturated with monovalent base gives a lower heat of wetting than one with a divalent base.

(3) Moisture absorption is highest with soil saturated with Ca and lowest with K. Sodium saturated soil occupies a peculiar position in as much as at high humidities its moisture absorption is the highest.

(4) Adsorption of methyl violet and crystal violet was greatly influenced by the nature of the replaceable ion so much so that Gedroiz declared the method quite useless for giving any indication of the degree of colloidility of a soil.

The above conclusions have been arrived at by working with soils in which all exchangeable bases have been replaced by a single one by treatment with a neutral salt. This treatment, as has been pointed out before[1], leads to the formation of Di-hydrogen acid saloid, or the neutralization of the clayic acid to H_1 stage, and since the soil can take up further amount of bases it was of interest to study the effect of the gradual introduction of different ions (up to the saturation point) on the absorption of ammonia and water as well as on heat of wetting.

EXPERIMENTAL.

A heavy black cotton soil (containing 60 per cent. clay) was completely desaturated by exhaustive treatment with N/20 HCl. The clayic acid thus produced was then gradually neutralized with different alkalies up to the "saturation" point (normal saloid formation) Li, Na, K, Am.hydroxides were used in the presence of excess of alcohol to avoid dispersion and facilitate filteration. The soils after treatment were filtered and dried. The filtrate in every case only contained traces of alkali except in the last of the series.

Various determinations on these soils are recorded below :—

AMMONIA ABSORPTION.

Two methods were used for studying ammonia absorption :—

(a) Leaving a weighed quantity of soil with excess of ammonia solution in the oven (100--105°C) overnight and determination of ammonia absorbed by distillation with lime.

(b) By passing ammonia gas over air dry soil contained in a conical flask for 5--6 hours and subsequently driving away the excess by passing air for 48 hours. Ammonia absorbed being determined as in (a).

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[1] Puri, A. N. Studies in Soil Colloids, part I. *Mem. Dep', Agri, India, Chem Ser.*, Vol. XI, No. 1 (1930).

The results given in Table 1-A and 1-B leave no doubt that ammonia behaves like any other base in neutralizing the soil acidoids, and when the base neutralizing power of a soil is partly or wholly satisfied by any other base, the amount of ammonia "absorbed" is considerably reduced and may even become nil. Therefore the method cannot be used for measuring the colloidal behaviour, though it could be used for measuring the saturation capacity of completely unsaturated soils.[1]

TABLE 1-A.

Ammonia absorption by Akola soil treated with varying amounts of hydroxides after the N/20HCl treatment. (Samples oven dried with excess of ammonia except in the case of ammonia soil which was left in the oven with water alone.)

M. eq. of exchangeable base per 100 grams of soil	Exchangeable ions. M. eq. of ammonia retained by various samples							
	Na	K	Li	NH_3	Ca	Mg	Sr	Ba
15	38·0	38·68	37·6	17·64	..	56·94	53·6	43·86
30	28·62	26·2	32·2	32·64	45·4	54·04	44·2	37·06
40	20·12	18·36	32·68	43·4	..	..	..	..
50	17·02	9·14	22·02	49·78	..	47·82	27·8	24·18
70	5·22	0	11·1	53·66	..	37·98	17·36	8·86
90	0·94	0	2·34	53·34	..	..	..	..
100	0·58	..	·76	54·18	13·4	29·0	9·88	3·62
120	0	..	·64	53·70	..	17·12	8·72	4·14

TABLE 1-B.

Ammonia passed over air dry soil for 5-6 hours ; air then passed for 48 hours.

M. eq. of exchangeable base per 100 grams of soil	Exchangeable ions. M. eq. of ammonia retained by various samples						
	Na	K	Li	Ca	Mg	Sr	Ba
15 . . .	70·8	59·0	62·6	..	66·4	64·4	54·8
30 . . .	56·8	43·90	56·8	54·7	64·4	42·8	37·8
40 . . .	46·0	33·2	36·2	..	..	..	..
50 . . .	37·6	24·6	30·0	..	60·3	39·6	..
70 . . .	21·4	7·50	17·0	..	54·2	26·0	16·4
90 . . .	9·4	1·2	14·3	..	..	..	..
100 . . .	5·4	0·6	14·4	28·1	46·0	15·70	15·4
120 . . .	2·8	0·4	15·3	..	41·3	13·2	12·0

[1] Puri, A. N. Studies in soil colloids, Part V, Methods of determining saturation capacity and degree of saturation of soil. *Mem. Dept. Agri. India, Chem. Ser.*, Vol. XI, No. 8 (1930).

HEAT OF WETTING.

Heat of wetting was determined in a heavily lagged Dewar tube. No attempt was made to apply the various corrections, but the conditions were kept strictly the same for all determinations, 10 gram portions of soils were first dried in the oven (100—105°C) and then kept in a desiccator over H_2SO_4 for a week in an ante-room where the variation of temperature was small. As the redistilled water used throughout was kept close by the desiccator containing soils, both had attained the same temperature and no correction was necessary for heat transference from soil to water or *vice versa*.

The rise of temperature produced by adding 10 grams of soil to 25 c.c. of water was recorded with a thermometer sensitive to ·002°C. Results given in Table II in terms of calories per grm. of dry soil confirm the conclusion arrived at by Pate (loc. cit.) that soil with a monovalent replaceable base gives a lower heat of wetting than one with a divalent base. A possible explanation of this may lie in the fact that alkali saturated colloids are very slow to wet (cf. Mattson[1]).

TABLE II.

Heat of wetting of soil affected by the nature and amount of replaceable base.

Replaceable base	Amount M. eq. per 100 grams	Heat of wetting, calories per gram
Li	15	6·94
	30	6·89
	50	5·98
Am	15	6·63
	30	6·49
	50	5·65
Na	15	7·00
	30	6·87
	50	6·60
K	15	6·54
	30	6·88
	50	5·62

[1] Mattson, S. The influence of exchangeable bases on the colloidal behaviour of soil materials, *Proc. First Int. Cong. Soil Sci.*, Washington (1928) Commission II, p. 185.

TABLE II—*contd.*

Replaceable base	Amount M. eq. per 100 grams	Heat of wetting, calories per gram
Mg	15	8·86
	30	7·90
Sr	15	8·43
	30	8·45
	50	8·40
Ca	30	9·76
	60	9·35
	100	9·21
Ba	15	10·09
	30	8·77
	50	8·04
H	....	8·72

It is clear from Table II that heat of wetting could not be used for measuring the absolute colloidal content of soils.

It might be mentioned that the above remarks only apply where a factor is used for computing the colloidal content of a soil from heat of wetting or NH_3 absorption data, or two soils compared as to their colloidal behaviour from such data. If, on the other hand, the formula $\frac{\text{Heat of wetting of colloid} \times 100}{\text{Heat of wetting of soil}}$ is used, there can be no serious objection beyond the one that with the newer methods of mechanical analysis when maximum dispersion could be attained with very little trouble (see for example the writer's NaCl—NaOH method) there is no justification for having recourse to a more tedious process which ultimately rests on the mechanical analysis.

MOISTURE ABSORPTION.

Absorption of water vapour from saturated atmosphere was the old method of determining hygroscopic co-efficient, the difficulties of which have been discussed by the author elsewhere[1]. For instance, it was shown that the "Hygroscopic Co-efficient" could not be satisfactorily determined, even when the technique was refined considerably beyond that reasonably possible in routine determinations.

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[1] Puri, A. N. *Journ. Agr. Sci.*, 15, p. 272 (1925).

Mitscherlich[1] has suggested that hygroscopicity of the soil should be determined by bringing it in equilibrium with the partial pressure of water vapour afforded by 10 per cent. sulphuric acid (96 per cent. humidity). Whereas the American Bureau of Soil workers recommend 3·3 per cent. H_2SO_4 (=99 per cent. humidity) for the same. Puri et al[2] suggested moisture absorption at 50 per cent. humidity as this occurred on the point of inflexion of the vapour pressure-moisture content curve and any error in controlling vapour pressure would only result in a small difference in equilibrium moisture content (Fig. 1).

Another difficulty about my moisture absorption experiments is the definition of " dry " soil. The usual practice is to dry in an oven at 100—105°C. This has the advantage of rapidity but much confidence cannot be placed in it. Drying over H_2SO_4 is to be preferred though the process is low. Both these methods are not infrequently attended with permanent changes in the soil colloids.

Typical Vapour pressure curve of soil (Harpenden common)
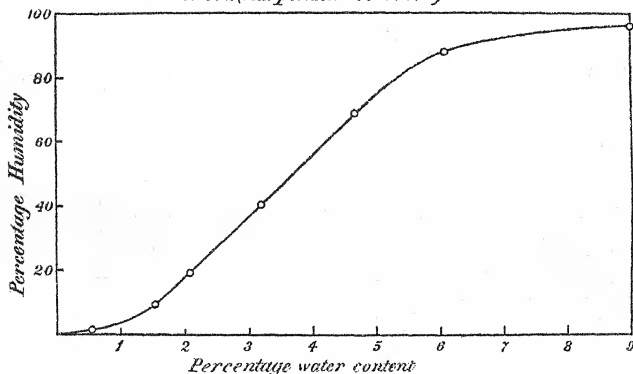
Fig. 1.

It will be seen from a typical vapour pressure curve for soil (fig. 1) that though smooth all along it is made up of 3 portions i.e., from 0 to 10 per cent., from 10 to 70 per cent., and from 70 to 100 per cent. humidity. The interval between 10 and 70 per cent. is sensibly straight and thus could be made the basis of expressing hygroscopicity of soils. The influence of the nature of the replaceable base is felt greatest in the portion up to 10 per cent. humidity and above 70 per cent. humidity. In

[1] Mitscherlich, E. A. und Floess, R. *Int. Mitt. Bodenk.*, Bd. 1, Heft 5, p. 463 (1912).
[2] Puri, A. N.; Crowther, E. M.; and Keen, B.A. *Jour. Agri. Sci.*, 15, p. 68 (1925).

the former case, it is due to hydration and in the latter, due to the flocculated or deflocculated state of the soil. It would seem, therefore, that the specific influence of replaceable ions could be partly eliminated by considering the soil in equilibrium with 10 per cent. humidity as "dry" from the point of view of hygroscopic moisture. In Table III are recorded the results of moisture absorption by H_2SO_4 dry soil samples (the same as used for heat of wetting) at 10, 50 and 70 per cent. humidity. The values for 70 per cent. humidity are also given on the basis of soil in equilibrium with 10 per cent. humidity as dry.

TABLE III.

Moisture absorption by soils containing varying amounts of exchangeable bases.

Exchangeable ion	Amount M. eq. per 100 grm.	EQUILIBRIUM MOISTURE CONTENT PER CENT.			10 per cent. humidity dry basis
		H_2SO_4 dry basis			70 per cent. humidity
		10 per cent. humidity	50 per cent. humidity	70 per cent. humidity	
Na	15	4·58	10·48	12·95	8·01
	30	4·57	9·96	12·50	7·59
	40	4·51	9·98	12·71	7·86
	50	4·62	10·06	12·50	7·62
	70	4·94	10·30	12·72	7·42
	90	5·07	10·02	12·35	6·93
	100	4·95	10·00	12·30	7·01
	120	5·46	10·23	12·62	6·78
K	15	4·00	9·76	11·69	7·48
	30	3·19	8·59	10·16	6·75
	40	2·95	8·32	10·03	6·87
	50	2·93	8·30	9·85	6·72
	70	2·70	7·72	9·30	6·43
	90	2·70	7·18	8·49	5·65
	100	2·59	7·12	8·61	5·87
	120	2·63	6·76	8·08	5·31

TABLE III—*contd.*

Exchangeable ion	Amount M. eq. per 100 grm.	EQUILIBRIUM MOISTURE CONTENT PER CENT.			
		H₂SO₄ dry basis			10 per cent. humidity dry basis
		10 per cent. humidity	50 per cent. humidity	70 per cent. humidity	70 per cent. humidity
Li	15	3·95	9·94	12·60	8·33
	30	3·70	9·44	12·22	8·22
	40	3·42	9·17	12·16	8·38
	50	3·25	8·68	11·67	7·98
	70	3·34	8·70	11·97	8·14
	90	3·34	8·52	11·53	7·93
	100	3·43	8·16	11·39	7·09
	120	3·40	8·15	11·33	7·67
NH₃	15	2·84	8·50	10·45	7·33
	30	2·71	7·67	9·53	6·64
	50	2·65	7·25	8·99	6·23
Sr	15	2·99	9·17	11·84	8·39
	30	2·80	9·4	11·95	8·91
	50	2·86	9·48	12·13	9·04
Mg	15	2·96	9·01	11·53	8·41
	30	3·14	9·10	11·64	8·04
	50	3·47	8·82	11·26	7·63
Ca	30	3·89	10·66	12·14	7·51
	60	4·12	10·87	13·23	8·36
	100	3·94	10·08	12·01	7·09
Ba	15	3·39	9·72	12·12	7·52
	30	2·86	9·10	11·48	7·27
	50	3·53	8·95	11·55	7·21
H	120	4·07	10·19	12·61	8·20
			9·1 mean		7·7 mean

Hygroscopicity of soils with
different exchangeable bases

● 70 % humidity 10 % dry basis.
○ 50 % humidity
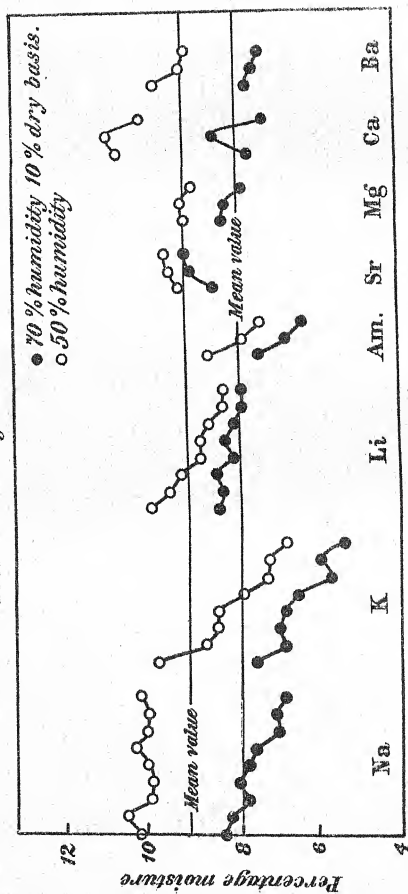
Increasing amounts of exchangeable ion

Fig. 2.

The results with 50 per cent. humidity (H_2SO_4 dry basis) as well as 70 per cent. humidity (10 per cent. humidity basis) are plotted in Fig. 2. It is seen that hygroscopicity on the whole decreases as the acidoid is neutralized and different ions have their specific influence in bringing about this change. The difference, though not very great, is in favour of adopting 10 per cent. humidity as the dry basis. The above results were confirmed with another soil series in which different ions had been introduced by exhaustive treatment with neutral salts. (Table IV).

TABLE IV.

Influence of replaceable ion on moisture absorption. (Soil from Hoosfield, Rothamsted.)

Replaceable ion	EQUILIBRIUM MOISTURE CONTENT PER CENT.	
	50 per cent. humidity (H_2SO_4 dry basis)	70 per cent. humidity (10 per cent. humidity basis)
H	3·94	2·60
Al	3·71	2·74
Ba	3·20	2·34
Ca	3·98	2·69
K	2·48	2·12
Na	3·19	2·29

There are several other advantages, besides the one mentioned above, in adopting 10 per cent. humidity equilibrium value as the " dry " basis for expressing hygroscopicity of soils. Lying on a straight portion of the vapour pressure curve it can be determined with greater precision than the H_2SO_4 dry point. On this basis the vapour pressure curve up to 70 per cent. humidity being a straight line passing through the origin, determination of a single value will enable us to interpolate hygroscopicity at any other humidity between 10 and 70 per cent. Further values of hygroscopicity given in this paper will refer to 10 per cent. humidity basis. Anderson and Mattson[1] have found a positive correlation between hygroscopicity, base exchange capacity, and $\frac{SiO_2}{Al_2O_3 + Fe_2O_3}$ ratio. However, it is easily seen that the relationship is only qualitative. In Table V are given values for hygroscopicity, and base exchange capacity for clay fractions (·001 mm. particles) from six different soils. The various fractions were first acid treated to eliminate the specific influence of exchangeable ions. The results only show a rough correlation.

---

[1] Anderson and Mattson. *U. S. Dept. Agri. Bull.* No. 1452 (1926).

TABLE V.

Hygroscopicity and base exchange capacity of clay fractions from different soils.

Clay fraction							Hygroscopicity 70 per cent. humidity	Base exchange capacity M. eq. per 100 grm.
1	.	.	.	.	.	.	3·21	23
2	.	.	.	.	.	.	5·07	31
3	.	.	.	.	.	.	5·31	32
4	.	.	.	.	.	.	6·63	41
5	.	.	.	.	.	.	4·60	5
6	.	.	.	.	.	.	2·68	2·6

We thus see that in order to define completely colloidal behaviour of a soil it is necessary to know its clay content, hygroscopicity and base exchange capacity : the first measures the size, second the surface development, and third the surface reactivity. Either one of these tells only a part of the story and together they constitute three of the most important " single values " to characterise the colloidal behaviour of a soil.

The first two of these values *i.e.*, the clay content and hygroscopicity can be determined with precision, but a good deal of confusion exists in soil literature as to the exact meaning of base exchange capacity, a full discussion of which forms the subject matter of Part V of this series (loc. cit.).

A word about dye adsorption already referred to. This method for measuring soil colloids though old has been recently advocated by American Bureau of Soils workers and is based on a grave misconception, brought about no doubt by the too frequent use of the term " adsorption ". It is to be remembered that only basic dyes are " adsorbed " by soils and the phenomenon is an example of base exchange so well known, and all the results obtained through dye " adsorption " could be reproduced with any neutral salt and much more precisely and quickly if the basic ion can be determined easily by analytical methods.

The shrinkage method of Tempany [1] needs only a brief mention. Tempany takes the total shrinkage occurring between a moisture content defined as the point of maximum plasticity and complete dryness. This value, he considers, measures the colloidal content of a soil. Haines has pointed out [2] that, if differences in porosity of the dry soil be neglected, the shrinkage so measured depends only on the moisture content at the point of maximum plasticity and hence a simple determina-

[1] Tempany : *Jour. Agri. Sci.*, 7, 312 (1917).
[2] Haines. *Ibid* 13, 296 (1923).

tion of moisture would suffice almost as well for making the comparison. As a "single value", it has probably the same significance as Keen's "sticky point"[1] for which no such claim is made.

The method of Van Bemmelen for determining the total quantity of inorganic colloidal matter in soils by chemical means (digestion with HCl and extraction with NaOH, etc.) is very old. On account of its tediousness it has not found much favour with the modern soil scientists. On the whole, it will probably give as good a correlation with other factors as the well known $\frac{SiO_2}{Al_2O_3 + Fe_2O_3}$ ratio of Mattson.

In view of the fact that a number of the proposed methods of measuring the colloidal content evaluate only certain phases of the colloidal behaviour of soils, it is better to adopt the very happy terminology of Keen and speak of them as "Single Values". This will clear a good deal of the confusion that exists as to the exact significance of these measurements.

Summary.

(1) Methods of determining soil colloids have been briefly reviewed. It appears that a number of such methods are affected by the nature of the exchangeable base in the soil and thus could not be used for the purpose they are intended for.

(2) It is suggested that hygroscopicity of a soil should be measured by bringing it into equilibrium with an atmosphere of 70 per cent. humidity; and soil in equilibrium with an atmosphere of 10 per cent. humidity should be considered as dry basis for referring to hygroscopic moisture.

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[1] Keen and Coutts. *Jour. Agri. Sci.*, 18, p. 740 (1928).

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Studies in Soil Colloids, Part V

Methods of determining Saturation Capacity and degree of Saturation of Soils

BY

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STUDIES IN SOIL COLLOIDS, PART V.

METHODS OF DETERMINING SATURATION CAPACITY AND DEGREE OF SATURATION OF SOILS.

BY

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(*Received for publication on 3rd November 1929.*)

The state of saturation of the soil (V) is defined by Hissink [1] as the ratio of the amount of absorptively bound bases in the soil (S) to the amount of bases the soil is capable of binding (T), both values being expressed in equivalents. Thus $V = \frac{100}{T} S$. The value T—S is the acidic hydrogen, that is the still "unsaturated hydrogen of the clay and humic acid". This is the most comprehensive and clear cut definition yet known. It is only in the experimental determination of T and S that the utmost confusion prevails in soil literature.

Hissink himself who compared ten different methods [2] found the degree of saturation for a single soil varying from 22 to 88. He came to the conclusion "that when referring to the degree of saturation it is always necessary to mention the method used".

This is also shown in a striking manner from the measurements of surface active hydrogen in a completely unsaturated soil (Akola) by various methods (Table 1). The soil was first treated with $\frac{N}{20}$ HCl, so that it could develop the maximum amount of surface active H.

TABLE I.

Method	Surface active H (M. eq. per 10 grams of soil).
Hopkins	[3·37
Daikuhara	[3·38
Loew	2·00
Jones	3·89

[1] Hissink, D. J. *Faraday Soc. Trans.* 20, 60 (1925).
[2] Hissink, D. J. Report on Soil absorption. *Proc. First Int. Cong. Soil Sci., Washington* (1927).

(133)

TABLE 1—*contd.*

Method	Surface active H (M. eq. per 10 grams of soil)
Kappen (single treatment)	3·87
Exhaustive treatment Ba acetate	5·67
Hutchinson and Maclennan	4·4
Veitch	4·72
Truog	5·00
Tacke	5·90
pH7	5·10
(S) Hissink	12·0
Gedroiz	4·4
Author's method to be described later	7·6
Excess Ba(OH)₂ added and washed for 3 months in CO₂ free atmosphere .	6·5

When variations of the order indicated above are confronted in the measurement of a certain factor, there must be something very seriously wrong with the principle underlying some of the proposed methods. In other words, they do not measure what they are supposed to.

In soils containing little $CaCO_3$, the determination of (S) value presents no serious difficulty; almost any one of the well known methods such as Hissink's, Gedroiz's, Kelley's, Bobko-Askinasi's could be used. The difficulty arises, however, on account of the fact that various workers have got a different conception of a saturated soil. For instance, Gedroiz thinks that, when a soil is exhaustively treated with N. $BaCl_2$ solution, its saturation capacity is reached and the degree of saturation is measured by the total amount of acid set free by the above solution. It has been shown in Part I of these studies that only H_1 is replaced by neutral salt reaction, a quantity which is approximately one-third the total amount of surface active hydrogen in a completely unsaturated soil. Gedroiz's method therefore cannot measure the degree of saturation of a soil, in other words, a soil that will be shown by his method as saturated may be very much unsaturated. Similar remarks apply to his " Volume of absorption " which is defined as the maximum quantity of cations that a soil is capable of taking up, but is measured by the total amount of ions (including hydrogen) that can be displaced from a soil (all quantities expressed in equivalents). For soils having no free H_1, Gedroiz's ." volume of absorption " is in fact identical with the S value as defined by Hissink. If a soil that is exhaustively treated with

BaCl$_2$ till no more acid can be displaced (saturated according to Gedroiz) is exhaustively treated with Ba acetate, a further lot of H can be displaced. The total amount of H thus replaced is equivalent to the total Ba taken up by the soil (determined after washing excess of Ba acetate and displacing Ba by HCl). This is shown in Table II where a number of completely unsaturated soils were thus treated.

TABLE II.

Showing the acid displaced by Ba acetate treatment after the BaCl$_2$ treatment.

Soil	MILL. EQUIVALENTS PER 10 GRAMS OF SOIL			
	H,BaCl$_2$	H, Ba acetate	Total H	Total Ba
Gonhri	·511	0·726	1·237	1·85
Rendzina	1·373	1·204	2·577	2·625
Hoos Fallow	1·357	0·900	2·257	2·28
Garden Humus	1·261	2·028	3·289	3·504
Gokak	1·676	0·676	2·352	2·410
Dharwar	4·588	1·612	6·200	6·358

Kappen uses Ca acetate to replace the surface active hydrogen which displaces a part of H$_2$ (provided the treatment is exhaustive) since Ca acetate has a higher pH than BaCl$_2$ solution. In fact, we can state a general rule *that the higher the pH value of a salt solution, the greater will be the absorption of its basic radicle by a given soil in contact with it.* This rule follows, as a matter of course, from the fact that the neutralization of clayic acid takes place at pH 11 or thereabouts; just as the neutralization of phosphoric acid takes place at pH 12.

It would seem from the above remarks that Hissink's method gives the nearest value to the total surface active H; since he employs a strong solution of Baryta when the whole of the clayic acid is converted into its Ba saloid.

'Sigmond and di Gleria [1] have rejected Hissink's method as giving too high a value for maximum absorption. They found that the base absorbing reaction takes place in two phases; the equivalent point of the acidoid can be titrated almost instantly, but the absorbing complex, though already chemically neutral, can absorb further amounts of Ba(OH)$_2$ slowly reaching the maximum of absorption after about 3 days interaction. It was considered that the end point of the instantaneous reaction (*i.e.*, their equivalent point) would give a truer measure of the point of saturation with bases at which the acidoid character of the absorbing complex is

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[1] *Proc. First Int. Cong. Soil. Sci. Washington* Vol. II, p. 155 (1928).

just neutralised. The method proposed by these authors is to add increasing amounts of $\frac{N}{10}$ Ba(OH)$_2$ solution, to the soil suspension and measuring the conductivity after each addition. The saturation capacity is determined by extrapolating the resulting conductometric titration curve. Of course, the same result could be obtained by adding increasing amounts of Ba(OH)$_2$ to the soil suspension, shaking for 2—3 minutes, filtering immediately and determining the amount of Ba(OH)$_2$ absorbed, just as in the case of Hissink's method. The main difference between the above two methods lies in the time of reaction which is 3 days according to Hissink and only few minutes according to 'Sigmond and di Gleria.

A moment's reflection will show that the adoption of this instantaneous reaction as the end point is not logically sound ; because if any base is added to a soil according to the saturation point determined by this method, that same soil will be unsaturated after three days and still more unsaturated after a week or two. As in nature we are dealing with bases which have reached equilibrium with respect to the soil complex, we cannot represent the degree of saturation with reference to the maximum absorbing capacity reached through a reaction that does not represent a state of equilibrium. The statement that the instantaneous reaction is the equivalent point and not the other, cannot stand in view of the following consideration. If the instantaneous reaction represents chemical saturation and the slow reaction mere absorption it is inconceivable that any base already saturating chemically the acidoid should be taken up by the absorbing complex leaving the former less saturated, since the chemical saturation must represent a stronger combination than the absorption saturation, yet this is what actually happens. The pH value of a soil after the addition of a base (however small a quantity) slowly decreases during the course of a few days showing that the product of combination is slowly passing to the less and less hydrolysable state. This phenomenon can be demonstrated very easily by adding any alkali to an acid soil suspension, in the presence of Brom Thymol Blue and some BaCl$_2$ to keep the suspension flocculated, till a permanent blue colour is obtained on leaving the suspension to settle for a few minutes. If this suspension is kept for a few days with occasional shaking, the blue colour turns green and goes back to yellow in 3 to 5 days showing the progressive decrease in the pH value. This phenomenon may be due to a slow diffusion of the base into the interior of the particles.

The main objection against Hissink's method is that the saturation point is reached in the presence of a high concentration of the base and represents a state that is *never* reached in nature and by giving too high a value for (V) is apt to be misleading from the practical point of view.

There is another important point which has been hitherto unrecognized *i.e.*, the base taken up by a soil to bring it from a lower to a higher pH is always considerably more than the base given up on taking it back from the higher pH to the original lower pH. This will be clear from Table III wherein are recorded the amounts of alkali required to take the soil from pH 7 to pH 10 and then the acid required to bring the pH back from 10 to 7,

TABLE III.

Showing the amount of alkali required for taking the soil from pH 7 to pH 10 and the acid required to bring it back to pH 7.

Soil	M. EQUIVALENTS PER 100 GRAMS OF SOIL	
	NaOH pH 7 to pH 10	Acid pH 10 to pH 7.
M. E. 10	20·5	7·5
Pod-soil No. 3	13·0	6·0
M. E. 9	24·0	7·5
M. E. 3	12·5	7·0
Clay soil Allepy (All the soils were first made completely unsaturated).	24	8·5

It is clear from Table III that for any given soil the pH value cannot be an index to its state of saturation, because the same soil at the same pH may have very different S and V values depending upon the previous history as to whether that particular pH was reached from a higher or lower initial value. Consequently, titrating a soil to any particular pH value and calling that the saturation point may prove very misleading.

In order to fix our attention on the maximum base absorbing capacity of a soil, we must first of all consider what happens to soils in nature. There are two most important factors working in nature which decide the state of saturation of a soil; namely, the weathering and leaching, the former causing the bases to accumulate, the latter tending to make the soil unsaturated. The equilibrium state (dynamic) of these opposing forces decides the degree of saturation of a soil.

If base is added to a soil equivalent to Hissink's saturation point, a large proportion of the former can be easily washed away. This easily hydrolysable part can never exist in nature and it would be just as well if we left that out of account. We may therefore define *the saturation point as the amount of base held by the soil which cannot be leached easily.* It could be determined by adding excess of Ba(OH)$_2$ to a soil and then leaching it. 'Sigmond and di Gleria (loc. cit.) have outlined such a method which consists in treating the soil successively with N. Ba(OH)$_2$? until complete replacement of the cations is indicated; the soil being then washed with BaCl$_2$ solution followed by leaching with water. The absorbed Ba is then determined by replacing with ammonium nitrate solution.

The above method is not only time consuming but it cannot be used for measuring the degree of saturation because even a fully saturated soil will absorb large

amounts of Ba, displacing other bases and the subsequent determination of Ba by ammonium murate replacement cannot measure (T—S) as in the case of Hissink's method but T. The two methods are quite different and can give approximately similar values only with fully unsaturated soils (S=0). The object of this investigation was to devise a simple method of determining S & T values which could be used for characterising soils in their genetic classification. The method finally adopted was as follows :—

To 10 grams of the soil 100 c.c. of water is added and shaken for about 2 hours with 2 grams of $CaSO_4$. 100 c.c. of $\frac{N}{5}$ $Ba(OH)_2$ is then run in and the mixture shaken for about 6 hours during a couple of days. 10 drops of phenolphthalein solution added and $\frac{N}{10}H_2SO_4$ run into it till the colour of the supernatant liquid is discharged. The acid is then added gradually shaking the suspension vigorously after each addition. The titration is complete when absolutely colourless supernatant liquid is obtained on leaving the suspension for a couple of hours. At first the pink colour slowly reappears when more $\frac{N}{10}$ H_2SO_4 is added. The titration is completed in about 2-3 days. The total Baryta minus the total acid gives the amount of base retained by the soil and is equal to (T—S). (T) is determined by washing another 10 grams of soil with $\frac{N}{20}$ hydrochloric acid till all the replaceable bases are removed (tested by the absence of Ca ions in the filtrate) followed by washing with water till the free acid is removed. The soil is then transferred to a bottle and back titrated after adding excess of $Ba(OH)_2$ exactly as described above. From the values of T and (T—S), (S) is easily known when V can be calculated by the formula $V=\frac{100 S}{T}$. The method is very simple and rapid as even 20 soils can be started all at once. The alkali or the acid is added to one, while the others are settling. The titration is carried out best in 500 c.c. reagent bottles with rubber corks.

It should be noted that in the method outlined above although $Ba(OH)_2$ is used, the whole of it is converted into $Ca(OH)_2$ and the absorption of the base takes place in a saturated solution of lime and the neutralization of the latter is conducted in a saturated solution of $CaSO_4$. The method on account of the limited solubility of both lime and $CaSO_4$ is independent of the soil water ratio, and the experimental conditions remain the same whatever the nature of the soil. Of course, a weighed quantity of lime could be used instead of $Ba(OH)_2$ for the preliminary absorption, but the former has to be carefully ignited and weighed whereas a measured quantity of the latter could be added with ease. It is better to run a blank under identical conditions for ascertaining the exact strength of $Ba(OH)_2$ used.

(S) can also be determined by Bobko-Askinasi method, *i.e.*, the soil is leached with N. $BaCl_2$ followed by washing with water. Ba being displaced by N. HCl and determined ; or it could be determined by Kelley's method, *i.e.*, by displacing all the bases with ammonium chloride, leaching the excess of the latter and determining the ammonia retained by distillation with lime.

One important point has to be borne in mind. Whichever method is used for determining (S), the amount of free acid, if any, must be subtracted from the value thus obtained. This, of course, occurs only with acid soils (having free H_2).

The T value for a completely unsaturated soil (*i.e.*, after the acid treatment) can also be determined as follows :—The soil after the acid treatment is air-dried, then kept in a flat dish over N. ammonia contained in a dessicator for 48 hours. It is then transferred into another dessicator containing 90 per cent. H_2SO_4 and kept for 48 hours under vacuum.

It will be recognized that the NH_3 method is identical with the $(Ba(OH)_2$—$H_2SO_4)$ method. In both cases alkali is added and the excess removed or neutralized. In Table IV are given (T) values for a number of completely unsaturated soils both by $(Ba(OH)_2$—$H_2SO_4)$ and NH_3 methods and show a good agreement. Their clay content as well as hygroscopicity (determined after the acid treatment) are also recorded for comparison.

TABLE IV.

(*T*) *values for completely unsaturated soils by* $(Ba(OH)_2$---$H_2SO_4)$ *and* NH_3
method.

Soil						Clay per cent.	Hygroscopicity (10-70 per cent. interval) per cent.	T $(Ba(OH)_2$—$H_2SO_4)$	T (NH_3)
								M. eq. per 10 grams soil	
A. T. 5	.	.	○	.	.	16·5	1·66	2·4	2·3
A. T. 6	.	.	.	.	.	5·7	0·50	1·0	0·8
A. T. 7	.	.	.	.	.	7·3	0·64	1·6	1·6
A. T. 9	.	.	.	.	.	43·5	3·26	2·1	2·3
A. T. 10	.	.	.	.	.	34·2	5·45	3·5	3·5
A. T. 13	.	.	.	.	.	5·8	1·70	2·2	1·5
A. T. 15	.	.	.	.	.	18·4	0·57	0·9	0·8
A. T. 16	.	.	.	.	.	37·2	2·67	3·0	3·0
A. T. 17	.	.	.	.	.	36·2	2·11	4·2	4·2
A. T. 19	.	.	.	.	.	29·9	1·73	1·7	1·6
A. T. 20	.	.	.	.	.	5·1	0·38	0·8	0·6
A. T. 21	.	.	.	.	.	58·2	..	5·5	5·5
A. T. 23	.	.	.	.	.	60·6	7·27	7·9	7·6
A. T. 24	.	.	.	.	.	60·5	6·95	5·6	8·5 ?
A. T. 26	.	.	.	.	.	15·2	0·91	1·1	1·0
A. T. 27	.	.	.	.	.	40·4	1·28	2·3	..
A. T. 28	.	.	٬	.	.	67·3	4·35	3·1	3·1
A. T. 29	.	.	.	.	.	34·5	1·87	1·8	1·5

B

The NH_3 method appeared to possess an attractive simplicity and it was considered at first that the method could be directly used for measuring (T—S), but this supposition was not realized in practice. It is probable that a part of ammonia displaces the base already present in the soil which results in too high a value being obtained for (T—S). This view is supported by the fact that the weaker the base already present in the soil, the greater the discrepancy between the two methods. This is shown in Table V which records (T—S) values for soils having different values of (S). It is also clear that the $(Ba(OH)_2$—$H_2SO_4)$ method is independent of the nature of the exchangeable base.

TABLE V.

(T—S) values for partly unsaturated soils.

Exchangeable base	S M. eq. per 10 grams	(T—S) M. EQ. PER 10 GRAMS	
		NH_3 method	$(Ba(OH)_2$—$H_2SO_4)$ method
Mg	1·5	6·64	6·2
	3·0	6·44	4·4
	5·0	6·03	1·4
Ca	3·0	5·47	4·8
	6·0	4·24	2·4
	1·5	6·44	6·2
Sr	3·0	4·28	4·8
	5·0	3·96	1·4
	1·5	5·48	6·2
Ba	3·0	3·78	4·8
	5·0	..	1·1

In Table VI are given S, T and V values for various soils determined by the method outlined above. Soils Nos. 2, 5 and 11 are definitely known to be acid in the ordinary usage of the term. It is a significant fact that (V) for all the three is below 50 per cent. Since $(Ba(OH)_2$—$H_2SO_4)$ method gives approximately neutralization value up to H_2 limit and since H_1=H_2, and also since the lime requirement of a soil from the practical point of view is satisfied when it is neutralized up to H_1 limit (*cf*. Liming experiments in Part I of these studies), it is conceivable that soil will need lime when V falls below 50 per cent.

TABLE VI.

T, S and V values for various soils by the proposed method.

No. Soil	M. EQ. PER 10 GRAMS OF SOIL				(V) per cent.
	Clay per cent.	(T—S)	(T)	(S)	
1 M. E. 10 . . .	56·2	0·2	3·85	3·65	94·7
2 Podsol . . .	24·9	1·45	2·35	0·90	38·3
3 M. E. 9 . . .	28·6	0·2	4·4	4·2	95·4
4 M. E. 3 . . .	31·4	0·3	2·2	1·9	86·4
5 Kuppapuram . .	40·8	2·7	4·4	1·7	38·6
6 M. E. 2 . . .	26·5	0	0·96	0·96	100
7 M. E. 7 . . .	60·5	0	5·65	5·65	100
8 Badob . . .	66·5	0	5·71	5·71	100
9 M. E. 4 . . .	14·2	0·37	1·36	0·99	72·8
10 Gurdaspur . . .	..	0·22	1·06	0·84	79·2
11 Dacca . . .	35·0	1·32	1·84	0·52	28·3

These studies are being persued with a number of other soils.

SUMMARY.

1. Methods of determining saturation capacity and degree of saturation of soil colloids have been critically examined.
2. A new method of finding the state of saturation of soils with respect to bases has been outlined.
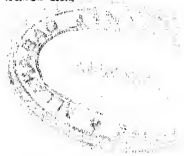
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